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The Journal of the SOCIETY OF DYERS AND COLOURISTS

Volume 68 Number 9

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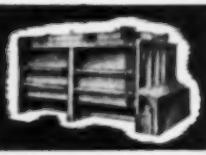

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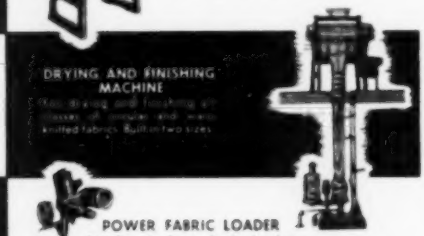

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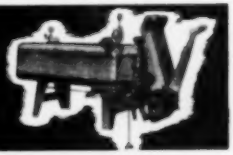

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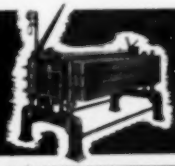

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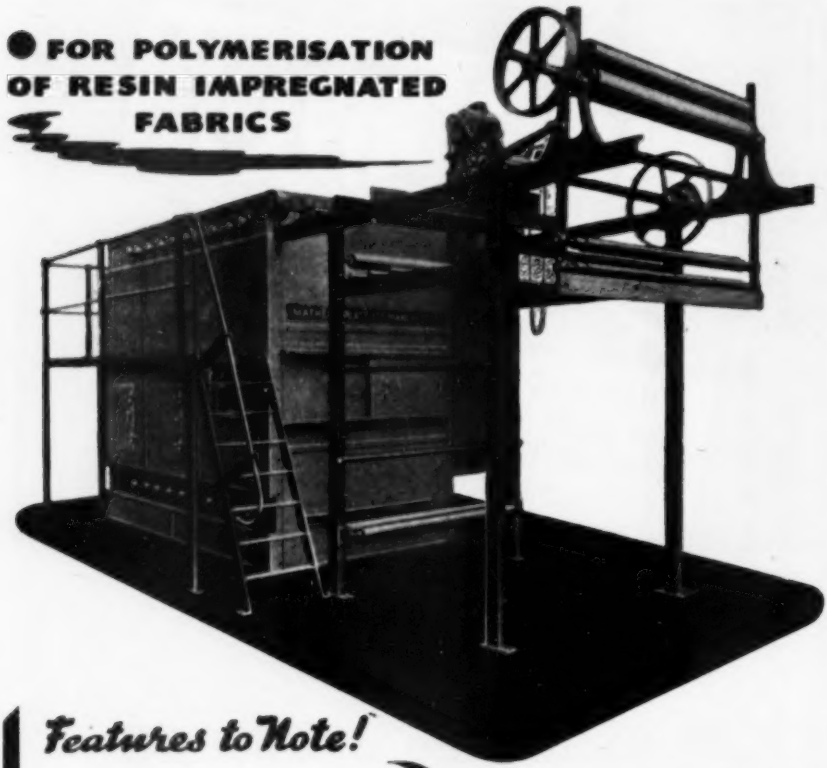

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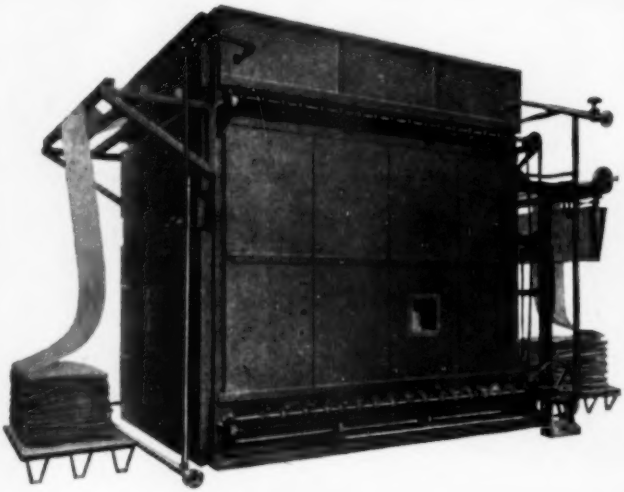

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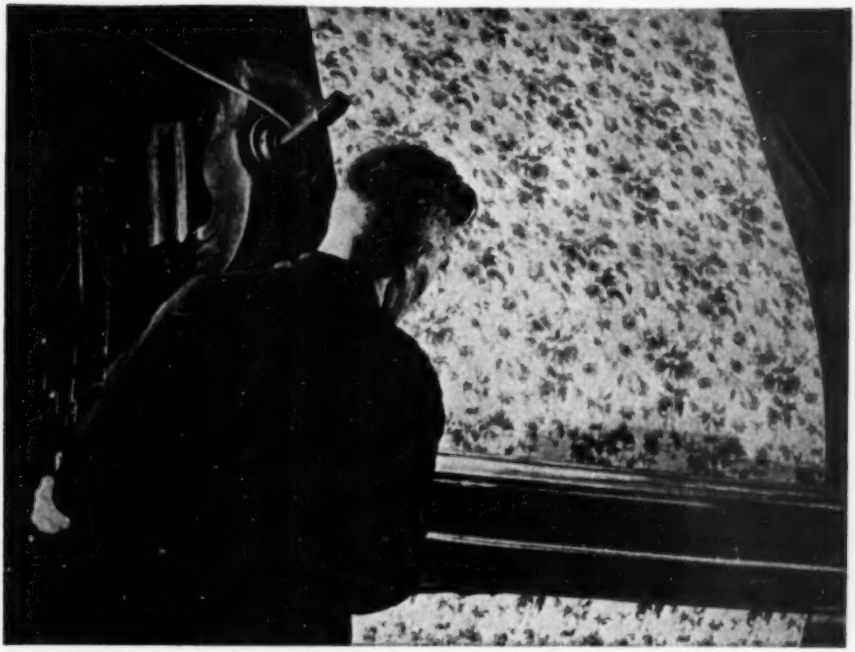

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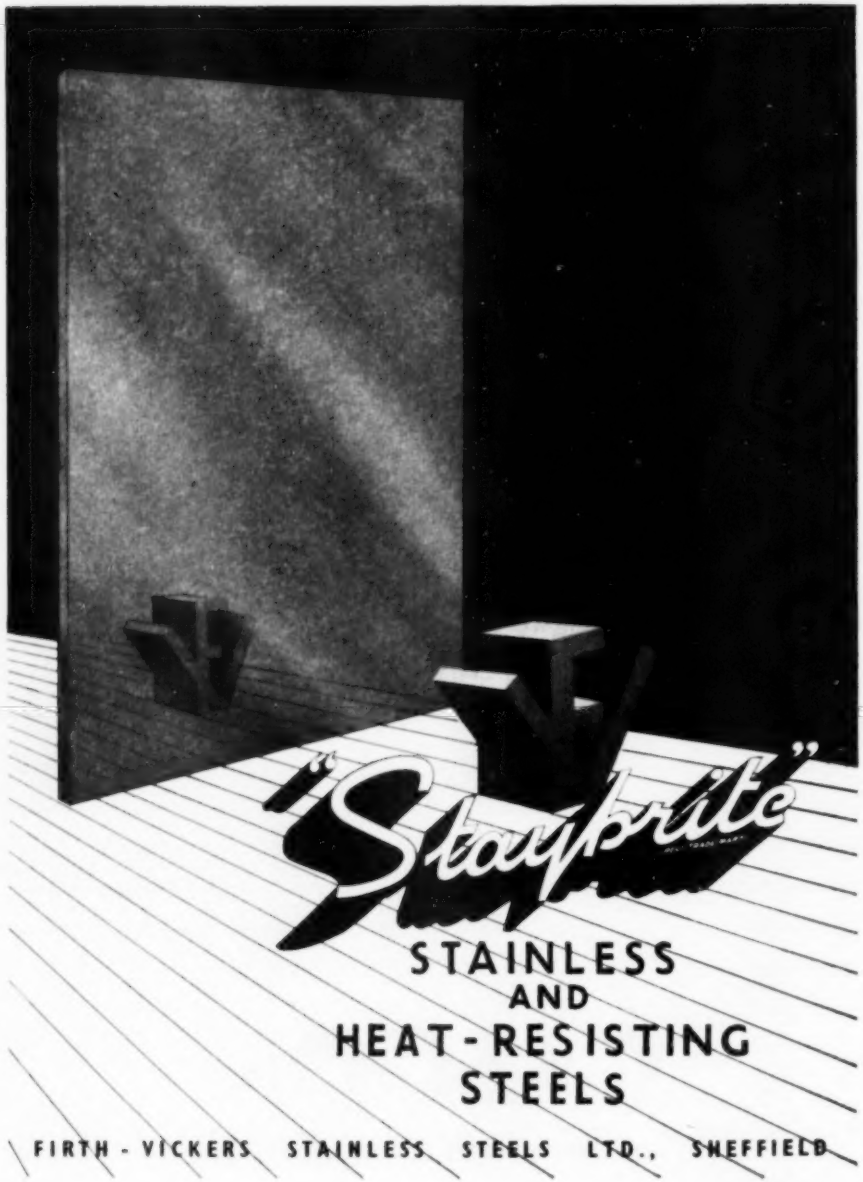
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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1952 and pages 237–240 of the July 1952 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Paper

The following paper has been accepted by the Publications Committee, and will appear in a future issue of the *Journal*—

LECTURE

A New Method of pH Control in Dyeing and Some Observations on the Metachrome Process

R. J. Hannay, W. H. Major, and R. Pickin

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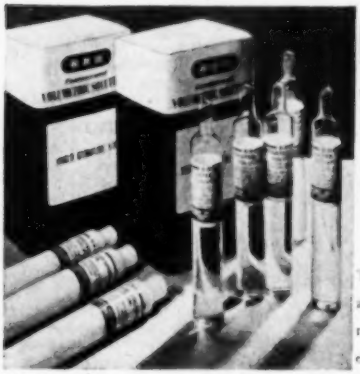

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Forthcoming Meetings of the Society

Society Bye-Laws, Rule 39 —

"Every member of the Society shall be entitled to attend the Scientific Meetings of the Society and, unless otherwise provided, each member may introduce not more than two visitors."

LONDON SECTION

All meetings held at 6 p.m. in the Rooms of the Royal Society, Burlington House, Piccadilly, London W.1, unless otherwise stated

1952

Friday 3rd Oct. — E. Atherton, Esq. (I.C.I. Ltd.). *The Prediction of Colour Matching Recipes*

Friday 7th Nov. — J. A. Radley, Esq., M.Sc.(Lond.), F.R.I.C. *Fluorescent Effect* (there may be a slight amendment to this title)

Friday 5th Dec. — Dr. P. R. Peacock (Royal Beatson Memorial Hospital, Glasgow). *Food Dyes*

1953

Friday 9th Jan. — *Uses of Microscopy in Textile Dyeing and Finishing* (Clayton Aniline Co. Ltd.).

Friday 6th Feb. — F. Ward, Esq. (Courtaulds Ltd.). *Methods and Principles of Textile Fireproofing*

Friday 6th March — Title to be announced later

MANCHESTER SECTION

Unless otherwise stated meetings are held in the Textile Institute, 10 Blackfriars Street, Manchester 3, and commence at 6.30 p.m.

1952

Friday 19th Sept. — Lecture to be announced later

Friday 17th Oct. — A series of short papers on *Radioactive Isotopes in Textile Technology*. Details of lectures to be announced later

Thursday 23rd Oct. — Dr. R. L. Wormell. *Researches on Regenerated Protein Fibres* (Joint meeting with the Textile Institute, Lancashire Section)

Wednesday 12th Nov. — L. Biggins, Esq., M.Sc. (I.C.I. Ltd., Dyestuffs Div.). *The Dyeing of Effect Threads*. Nicolson Institute, Leek

Friday 21st Nov. — LADIES' EVENING. Manchester College of Technology, commencing at 7 p.m. *Penny Plain. Twopence Coloured.* Lectures and demonstrations on the miscellaneous applications of dyestuffs. Speakers to be announced later

Friday 19th Dec. — Lecture to be announced later. Details to be announced later

1953

Friday 16th Jan. — G. G. Taylor, Esq. (Clayton Aniline Co. Ltd.). *The Measurement of Colour*

Friday 20th Feb. — W. Speke, Esq., B.Sc. (I.C.I. Ltd., Dyestuffs Div.). *High Temperature Dyeing of Viscose Rayon*

Friday 13th March — Manchester College of Technology. Afternoon and Evening Symposium on *New Fibres*. Details to be announced later

Friday 20th March — Joint meeting with the British Association of Managers of Textile Works. Midland Hotel, Manchester. Lecture by Dr. Fargher (Shirley Institute). Details to be announced later

Friday 17th April — ANNUAL GENERAL MEETING. Details of lecture to be announced later

Continued on page xxxv

MIDLANDS SECTION

Except when otherwise stated, Lecture Meetings commence at 7 p.m.

1952

Wednesday 1st Oct. — Discussion on Automatic Dyehouse Controls. F. Blezzard, Esq., *Design and Application*. G. Robinson, Esq., *Some Works Experience*. King's Head Hotel, Loughborough

Wednesday 29th Oct. — G. H. Lister, Esq., Ph.D., B.Sc. *Some New Angles on Detergency*. College of Technology, Leicester

Wednesday 19th Nov. — A. E. Stubbs, Esq., Ph.D., F.R.I.C. *Developments in Cotton Bleaching at home and abroad*. Victoria Station Hotel, Nottingham

Saturday 22nd Nov. — LADIES' EVENING — DINNER DANCE Black Boy Hotel, Nottingham

Wednesday 17th Dec. — *Colloquium*. Short papers on practical problems given by Local Members. King's Head Hotel, Loughborough

1953

Wednesday 28th Jan. — J. Boulton, Esq., M.Sc.(Tech.), F.R.I.C., F.T.I. *Modern Dyeing Theory for the Plain Man*. Masonic Hall, Hinckley, 7.30 p.m. (Joint meeting with the Textile Institute and Hinckley Textile Society)

Thursday 19th Feb. — E. M. Walker, Esq., F.T.I., F.R.M.S. *The Chemist and Technologist in the Hosiery and Knitted Goods Industry*. (Joint meeting with the Textile Institute). Carpet Trades Canteen, Kidderminster, 7.30 p.m.

Wednesday 25th Feb. — E. R. Wiltshire, Esq., B.Sc. *Azoic and other Fast Dyes on Knitted Cotton Fabric*. College of Technology, Leicester

Friday 13th March — MIDLANDS SECTION DINNER. Royal Hotel, Leicester

Wednesday 18th March — G. H. Osborn, Esq., F.R.I.C., A.M.Inst. M.M. *Colour in Chemistry* (Joint meeting with the British Association of Chemists). School of Arts and Crafts, Derby

Wednesday 22nd April — ANNUAL GENERAL MEETING OF THE SECTION. Gas Board Theatre, Nottingham

Towards the end of the session Dr. H. White (Textile Research Institute, Princeton, U.S.A.) will lecture in Nottingham. Particulars later

NORTHERN IRELAND SECTION

All Meetings to be held in Queen's Hotel, Belfast at 7.30 p.m.

1952

Wednesday 8th Oct. — DISCUSSION EVENING — *Brains' Trust*

Wednesday 12th Nov. — J. C. Brown, Esq. and G. G. Taylor, Esq. (Clayton Aniline Co. Ltd.). *Microscopy for the Dyer and Finisher*

Wednesday 10th Dec. — A. S. Cluley, Esq. (Courtaulds Ltd.). *Some Observations on Practice in the Dyeing and Finishing of Rayon Materials* (Joint meeting with Textile Institute)

1953

Wednesday 14th Jan. — E. R. Wiltshire, Esq. (I.C.I. Ltd.). *The Control of Azoic Dyeing Processes* (Joint meeting with Foremen Dyers Guild)

Wednesday 11th Feb. — F. H. Marsh, Esq. (Longclose Engineering Co. Ltd.). *Pressure Dyeing and Bleaching with Possible Indication of New Developments*

Wednesday 11th March — Dr. B. C. Gee. *The Educational Side of Textile Processing*

March/April — ANNUAL GENERAL MEETING and DINNER (date will be announced later)

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THE JOURNAL
OF THE
Society of Dyers and Colourists

Volume 68—Number 9 SEPTEMBER 1952 *Issued Monthly*

Proceedings of the Society

Recent Developments in Bleaching with Hydrogen Peroxide

W. S. Wood and K. W. Richmond

Meeting of the London Section held in the rooms of the Royal Society, Burlington House, Piccadilly, London, on 2nd November 1951, Mr. C. C. Wilcock in the chair

HYDROGEN PEROXIDE

In the past few years the application of hydrogen peroxide has been appreciably widened, and no doubt the wartime development of the use of high-strength solutions of hydrogen peroxide has stimulated a great deal of interest, both academic and practical. Its method of manufacture has been carefully studied, particularly in this country, and as a result, the economics of production has been improved. Unfortunately, present-day inflation has neutralised much of the reduction in the cost of manufacture, but when compared with other materials the increase in the cost of hydrogen peroxide since the war has been quite modest.

To-day hydrogen peroxide is produced essentially by an electrochemical process, and the old method based on the reaction of barium peroxide with an acid has been almost entirely eliminated. The electrochemical process depends upon the electrolysis of either a solution of sulphuric acid alone, a solution of ammonium sulphate in sulphuric acid, or a solution of ammonium sulphate and potassium sulphate in sulphuric acid. After electrolysis, the solution of persulphuric acid or persulphate is hydrolysed, whereby hydrogen peroxide vapours are obtained which can be fractionated directly to give solutions of, say, up to 50% H_2O_2 by weight. The technique of the distillation process has been so improved that the purity of hydrogen peroxide sold to-day is appreciably better than that sold immediately before the war, whilst the stability of the product is such that under ordinary conditions of storage either in this country or elsewhere this factor no longer presents any serious problem.

It is possible to manufacture on a large scale hydrogen peroxide of a purity comparable with that of distilled water, but this involves an additional operation, the expense of which is not normally justified for the textile field.

However, 90% (by weight) hydrogen peroxide (414 vol.) of this high grade of purity is available, and provided proper precautions are taken it can be handled quite safely in large quantities. Such concentrated material does not appear to have found direct application in the textile field, although a patent [1] has been taken out for rendering wool non-shrinking by the use of peracetic acid, and refers to a method for making the peracetic acid using high-strength hydrogen peroxide.

The interest in hydrogen peroxide has encouraged a more thorough investigation of its physical properties, and some of the results of this may be of value to the textile user. For example, it had long been held important to have appreciable quantities of acid present if the hydrogen peroxide were to be stable. This has been shown to be unnecessary (Fig. 1). The curve indicates relative rates of oxygen evolution from pure hydrogen peroxide at 100°C. over the pH range 2–7. The rate of oxygen evolution varies little below pH 6, so that the stability does not depend upon the presence of appreciable quantities of acid.

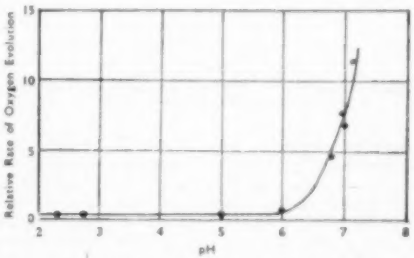

Fig. 1.—Effect of pH on Stability of Hydrogen Peroxide at 100°C.

BLEACHING

The literature on bleaching with hydrogen peroxide is extensive, and some idea of the scope of its application to textile fibres as well as its relation to other bleaching agents may be obtained

from the review of bleaching processes undertaken by the A.A.T.C.C.[2] Further, a bibliography on hydrogen peroxide bleaching alone covering the period 1930–50 has been prepared by Laporte Chemicals Ltd.[3] A detailed study of the literature reveals that the main consideration throughout has been the practical side of the bleaching operation, and the procedure appears to have been built up on that indicated in the first bleaching patent, taken out by Lebouteux in 1878 for bleaching silk [4].

The decomposition of hydrogen peroxide is fundamental to the bleaching process, and extensive study has been made of its kinetics in the past few years, particularly in the presence of catalysts. A relationship which may be of interest in the bleaching field is the rate of decomposition of hydrogen peroxide in the presence of an iron salt, measured at different pH values of the solution (Fig. 2). A maximum rate of decomposition is

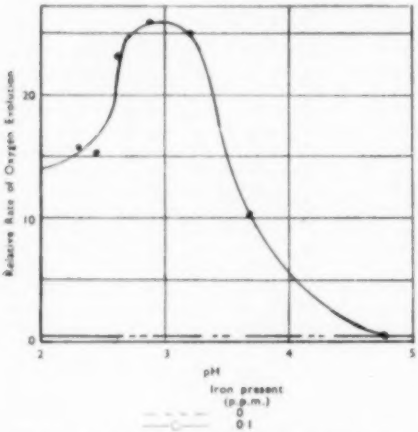

FIG. 2—Effect of pH on Stability of Hydrogen Peroxide at 100°C. in presence of Small Quantities of Iron

observed at pH 3. This, again, refers to a pure high-strength solution, although the same principle may be true for more dilute solutions, in which case the course of the reaction in bleaching systems may be affected by the presence of traces of other ions, e.g. catalysts such as iron from the stabilisers employed.

A somewhat complicated series of reactions is now used to explain the decomposition of hydrogen peroxide, and has supplanted the simple direct decomposition into a molecule of water and an atom of oxygen. The following series of reactions has been suggested to explain the decomposition of hydrogen peroxide in the presence of iron [5–7]—

$$H_2O_2 \rightleftharpoons HO_2^- + H^+ \quad \text{(i)}$$

$$HO_2^- + Fe^{3+} \rightarrow Fe^{2+} + HO_2 \quad \text{(ii)}$$

$$HO_2 + Fe^{3+} \rightarrow Fe^{2+} + H^+ + O_2 \quad \text{(iii)}$$

$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + OH^- + OH \quad \text{(iv)}$$

$$OH + Fe^{2+} \rightarrow Fe^{3+} + OH^- \quad \text{(v)}$$

$$OH^- + H^+ \rightarrow H_2O \quad \text{(vi)}$$

It may be of some significance that hydrogen peroxide and other peroxy compounds form free radicals in their systems, and it is suggested that the bleaching action could depend upon such radicals as HO_2 (equation ii) and OH (iv), rather than on oxygen itself.

Hydrogen peroxide is to-day used for bleaching all types of textile fibres—animal, vegetable, and synthetic. In this country, wool bleaching with hydrogen peroxide has probably received most attention, the application of hydrogen peroxide to cotton bleaching having been confined until recently mainly to the U.S.A.

The following examples have been chosen to illustrate recent developments in bleaching with hydrogen peroxide, but do not, of course, cover every aspect of the subject. The developments discussed in this paper include—bleaching of wool in acid solution, dyeing of wool after bleaching with alkaline solutions of hydrogen peroxide, bleaching of dark-coloured wools, continuous bleaching of cotton, simultaneous bleaching and dyeing of cotton and viscose rayon, bleaching of mixtures and unions, and applications of known methods in standard dyeing equipment.

WOOL BLEACHING DEVELOPMENTS

As there is doubt whether the simple oxygen evolution theory of bleaching can be sustained, it would be extremely valuable to have a picture of the mechanisms which control the bleaching operation, since such information might revolutionise bleaching techniques. If it is accepted that hydrogen peroxide can decompose into hydroxyl radicals in solution, then it is not impossible for the bleaching action to depend initially upon such hydroxyl radicals and proceed by a series of chain reactions involving perhaps not only decomposition of the hydrogen peroxide, but also reaction with impurities in the system and groups in the wool molecule.

Formation of hydroxyl radicals should be theoretically possible in acid solutions, thus supporting the idea that wool might be bleached in acid solutions of hydrogen peroxide. It is, of course, not new to suggest bleaching under acid conditions; in fact, as far back as 1897 a process was proposed for bleaching silk in an acid peroxide bath [8]. Also, in the period 1931–34 several patents were taken out for bleaching wool with hydrogen peroxide under acid conditions, in a solution containing, for example, an organic acid such as oxalic or formic acid [9–11]. However, as far as we know, and at any rate in this country, such conditions have not been applied to bleaching wool in any quantity.

The basic advantages of bleaching wool with hydrogen peroxide are as follows—the chemicals involved are not unpleasant to handle, conditions of operation are odourless, strengths of solutions are readily maintained, the process is safe and its operation is economical and labour-saving, the bleaching effect obtained on the wool is of high quality and permanent, and the material is also left in an odourless condition. Additional advantages which might be expected from acid bleaching conditions are—decreased chemical modification,

increased fastness of dyes, and improved handle and raising properties where such are called for. These properties are extremely valuable in wool processing, and considerable success has been achieved from all the above points of view.

In our original laboratory work undertaken to study acid bleaching, hanks of botany worsted yarn were immersed in solutions of hydrogen peroxide of 4 vol. strength containing a small fixed concentration of different acid radicals, whilst the pH values of the solutions were adjusted over the pH range 2–9 with caustic soda. After saturation the hanks were taken out, squeezed to remove excess liquor, and maintained in an oven at a constant temperature overnight. The samples were then dried and examined visually for bleaching. Various acids were used—sulphuric, phosphoric, formic, acetic, etc.—and the results were sufficiently encouraging to proceed with the investigation.

The rather surprising result was observed that the degree of bleaching varied little with pH, and there was not a great deal of difference between the acid-bleached samples and those on the alkaline side. The differences were so slight that classification by numerical grading was difficult. Under these simple conditions the kind of acid used also appeared to have little effect on the bleached result. The small differences which were observed appeared in the pH range 3–5 and at about pH 8, but these were noticeable only with one or two of the acids used. The results were somewhat qualitative in nature, and it is hoped to study them in greater detail later.

As some of the catalysts for hydrogen peroxide decomposition are found in sufficient quantity in most natural products (e.g. King [12] quotes iron figures in different varieties of wool of 30–100 p.p.m. as Fe), it is considered that the bleaching result may depend on the decomposition-pH relation for the catalyst present.

The immediate application of acid bleaching at the time of the work outlined above was found in blanket materials, and the authors are greatly indebted to Messrs. Charles Early & Co. Ltd., of Witney, for their willing and helpful co-operation in applying the results of the work to full-scale production conditions and for their help in developing the process to a satisfactory conclusion. An outline of the conditions now applied in the industry is given below.

The blankets are initially scoured, carbonised, milled, and neutralised. The hydrogen peroxide bleach bath is prepared at 4 vol. strength containing. 2¼ lb. of 85% formic acid per 100 gal. of solution and adjusted to pH 3-3·5 with soda-ash. The temperature is adjusted to 60–70°F.

The blanket material is saturated in the solution and passed through squeeze rollers, which return excess solution to the bath. The blanket material may then be piled and aged for, say, 24 hr. before drying.

The method is suitable for batch or continuous processing according to production requirements. In either case, the bleaching solution is maintained at its original concentration by regeneration, and may be used for bleaching a considerable quantity of material. Fig. 3 suggests one manner in which the cloth may be impregnated with the bleaching solution prior to ageing.

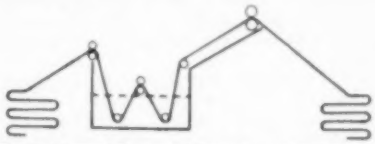

FIG. 3—Diagram of Suggested Plant for Bleaching Blankets

Experience has shown that under the best conditions of operation, particularly with efficient scouring, leaving the blanket pieces readily penetrable by the bleaching solution, the bleach obtained is as good as the standard expected when operating under alkaline conditions. One other advantage, which is, of course, an outcome of the method employed, is that impregnation is carried out in the bleaching solution at or about room temperature, and this represents an economy in steam compared with alkaline processes, which may operate at 120°F.

Quantitative figures are not yet available on the extent of modification undergone by wool treated by acid methods, and it is in fact doubtful whether any really satisfactory method of quantitatively assessing chemical damage of wool is known at present. However, the appearance and performance of the bleached blanket material during and after the raising operation indicated that the wool had not undergone undue damage. The bleached blanket material raised satisfactorily with the production of a full, springy pile.

Blanket materials containing coloured headings are regularly bleached under the conditions described, and the finished blankets are characterised by complete freedom from bleeding. It has also been observed in practice that many of the dyes tested are much more resistant to change in shade when bleached under acid conditions than under alkaline conditions.

The initial acid bleaching technique and its application to blankets has been considered at some length. Other recipes are being developed, and promising results are also being obtained in bleaching other forms of wool with acid solutions of hydrogen peroxide.

Medium-pigmented Woods

Hydrogen peroxide finds special application in bleaching prior to dyeing the medium-pigmented yellow East Indian woods which are mixed with lighter-coloured wool to confer springiness on yarn used in carpet manufacture. The value of hydrogen peroxide bleaching is to reduce the dark colour of the yarn and enable it to be dyed subsequently in light pastel shades. Such bleaching is at present carried out in alkaline solution and is therefore not a recent development. It does not appear to be widely appreciated, however, that a colour obtained by dyeing a bleached wool containing excessive concentrations of residual hydrogen peroxide can be inferior to that obtained on the unbleached wool.

Investigation of this problem in the laboratory has suggested that a residual hydrogen peroxide content up to 0·05% on the weight of wool might be tolerated to enable a normal dyeing process to be employed, although the use of minimum quantities of acid and low temperatures is recommended.

Table I illustrates the dyeing results obtained with varying concentrations of residual hydrogen peroxide, in a dyebath set with acid dye (0·05% on wt. of wool), Glauber's salt (10%), and sulphuric acid (5%). The liquor ratio was 30 : 1, and the weight of wool 28 g. The dyeing results are graded 1 to 5, where grade 1 represents the best result regarding true colour and depth of shade.

TABLE I

Bleaching Method	Hydrogen Peroxide (%)	Dyebath Temp. (°C.)	Grade
(i) Unbleached ...	0	100	3
(ii) Steeped and rinsed	0·01	100	1
(iii) Aged 48 hr. ...	0·08	100	1–2
(iv) Aged 48 hr. ...	0·08	90	1
(v) Aged 24 hr., rinsed ...	0·01	100	1
(vi) Aged 24 hr. ...	0·4	80	2–3
(vii) Aged 24 hr. ...	0·4	90	4
(viii) Aged 24 hr. ...	0·4	100	5

In order to overcome any difficulties in dyeing after bleaching, it is present practice to recommend that, following any of the usual alkaline methods of bleaching, the material be rinsed thoroughly in running water or subjected to not less than two rinses in clear water.

The results of these investigations agree in principle with, but differ in detail from, those of American investigators on the same problem [12].

Heavily Pigmented Wools

Fluctuations in wool prices and their general high level have been matters of great concern to the wool industry, and the question of employing cheaper forms of wool where possible has been a point of interest. Heavily pigmented wools can find a number of applications in admixture with the dearer, lighter-coloured wools if a reduction in intensity of colour can be effected. The depth of pigmentation and resistance to removal by bleaching vary considerably between different fibres, and it is not safe to predict that any particular method will bleach a particular sample of wool. In fact, experience has shown that on occasion very dark-coloured wools have responded to bleaching treatment much more readily than some of the less pigmented material. It must suffice to note that, whereas heavily pigmented wools do not in general respond satisfactorily to the normal alkaline bleaching methods, a variety of special methods have been developed, one of which involves application of the iron catalyst–hydrogen peroxide reaction already referred to.

COTTON BLEACHING

Perhaps the most outstanding development in the application of hydrogen peroxide to textile bleaching is to be found with cotton. Possibly as a result of the changing industrial conditions of the last ten years brought about by wartime necessities, the American manufacturers of hydrogen peroxide have taken the opportunity of developing their continuous bleaching process, and have achieved such satisfactory results that it is now claimed that more than 80% of the cotton bleaching in America is effected by this process. The success of this development appears to have originated in the application of direct steam heating to materials piled in a container, of which the J box is an outstanding example. Again, the availability of suitable materials of construction played an important part in its development. Although much has been written on continuous cotton-bleaching methods in the U.S.A., most of the published data refer to engineering design and chemical composition of solutions for plant operation, and little appears to have been written on the chemistry of the process.

Interest in continuous peroxide bleaching has now developed on the Continent. It has been claimed that the French have designed equipment to suit their own particular requirements, and that by the end of 1951 both France and Italy would have six operating units [14]. It appears to have been generally accepted, both on the Continent and in this country, that the American developments in continuous cotton bleaching were interesting for large-scale production but were not so attractive when applied to smaller quantities or to that type of business handling a variety of materials. In recent years, American manufacturers have become aware of the needs of the smaller textile concerns, and have introduced continuous plant for treating quantities of the order of 2–5 tons per day, compared with, say, 40 tons per day for some larger units.

In this country, there appears to be quickening of interest, and it may be that something concrete will emerge in the not too distant future. We have therefore carried out laboratory experimental work to determine the effect of varying the factors in the bleaching operation.

Cotton cloth was first scoured by a standard method using caustic soda, and after squeezing was immersed in solutions of hydrogen peroxide of various strengths, adjusted to pH values between 9·5 and 11·0 with sodium silicate ($3{\cdot}3\ SiO_2 : 1\ Na_2O$) and caustic soda. The relation between the time of immersion of the cloth and the take-up of hydrogen peroxide was determined under these varying conditions. The immersed material was then subjected to steaming for a given time, and its colour and fluidity were measured. The experimental steaming unit employed is shown diagrammatically in Fig. 4.

Fig. 5 shows that there is positive sorption of hydrogen peroxide from the aqueous solution, to an extent which varies over the pH range and decreases noticeably at the higher pH value. Thus, whilst a 100-g. sample of cotton taking up its own weight of liquid from a 2-vol. solution should theoretically contain 0·6 g. H_2O_2, the actual quantity ranged from 0·8 to 1·1 g. Further, sorption does not appear to be a critical function of time of immersion, as a comparison of the graphs

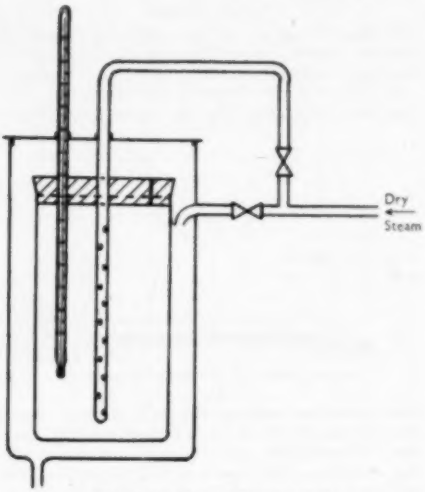

FIG. 4—Experimental Steaming Unit

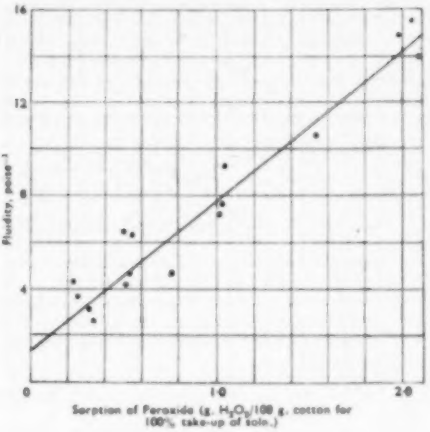

FIG. 6

shows little increase between 5 sec. and 15 min. Hence in practice passage of cloth through the bleaching solution may be as rapid as is normally desired.

Fig. 6 shows the relationship between take-up of hydrogen peroxide and fluidity of the bleached material. Fluidity is a measure of the average chain length of the cellulose molecules in cotton, low fluidity representing low chemical modification of the fibre, so that it may be inferred from the graph that, in order to cause minimum damage to the cotton, take-up of hydrogen peroxide should be kept to a minimum. Comparison of these graphs with those in Fig. 5 indicates that satisfactory fluidity figures are theoretically obtainable from the use of solutions of higher pH, in the region of, say, pH 11.

From our present standpoint, the most important factor to consider in cotton bleaching is the colour of the finished product, and Fig. 7 relates the whiteness of bleached cotton as measured in reflectance units to sorption of hydrogen peroxide. It is particularly interesting to note that reflectances of 94% (compared with a magnesium oxide standard as 100%), which are considered satisfactory, are obtainable by operation at pH 11.

Whilst these laboratory experiments are incomplete in themselves, the available information suggests that the necessary chemical conditions for the production of the best bleached result are also the most economical in operation.

American Methods

According to the published American methods, the cotton is passed through a caustic saturating tank (Fig. 8), from which it emerges through squeeze rollers adjusted to ensure the uptake of a predetermined weight of solution. The cloth then either (*a*) passes through a preheater, where it is subjected to the action of steam to raise its temperature very rapidly to about 212°F., and then passes into a J box or storage vessel; or (*b*) passes directly from the impregnator to the J box, into which steam is passed in order to raise and maintain the temperature.

The cloth is moved continuously through the J

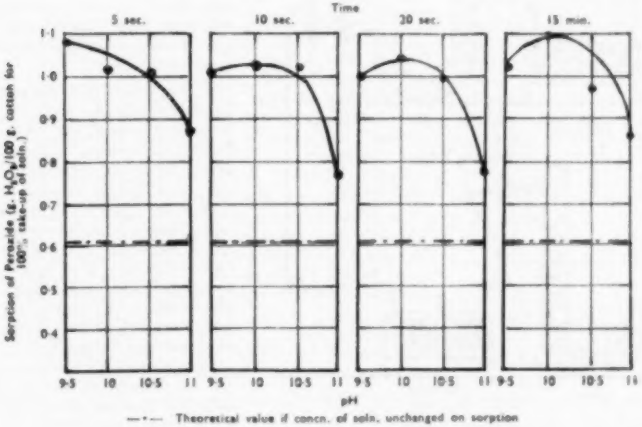
Time
5 sec.
10 sec.
20 sec.
15 min.
Sorption of Peroxide (g. H_2O_2/100 g. cotton for 100% take-up of soln.)
pH
—•— Theoretical value if concn. of soln. unchanged on sorption

FIG. 5

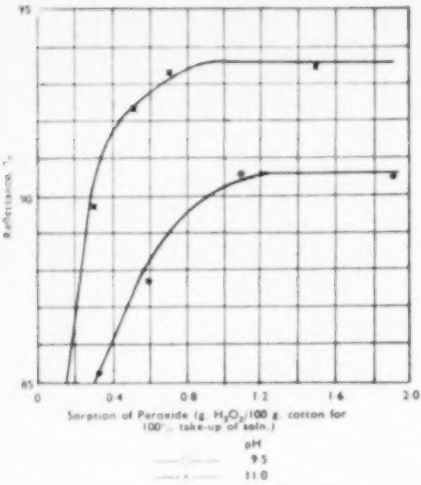

FIG. 7—Relation between Sorption and Reflectance at Various pH Values

box, which is of such a capacity that the cotton is subjected to the hot alkaline treatment for about one hour. The material then leaves the J box, and passes through a washer and into the second half of the unit, where it is impregnated with the bleaching solution and bleached by ageing for a similar time in a second J box before passing to the final wash and to the drier.

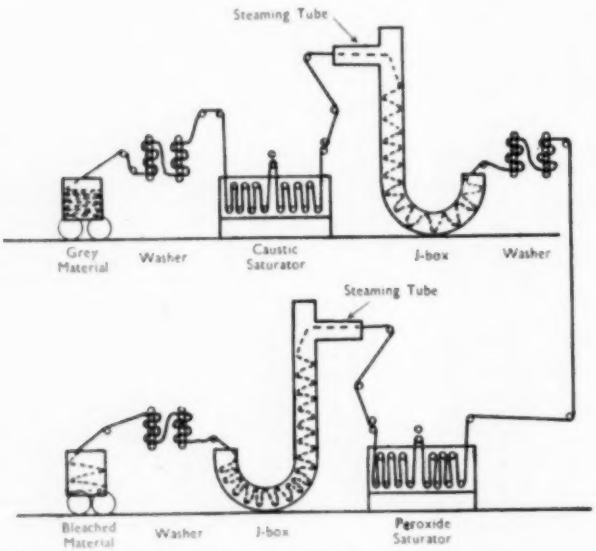

FIG. 8—Diagram of Continuous Bleaching Process

French Methods

Mouton[16] refers to two different types of machine, which are modifications of the American technique to handle smaller quantities of material.

(1) GANEVAL AND SAINT-GENIS MACHINE—The bleaching apparatus (Fig. 9) comprises a closed

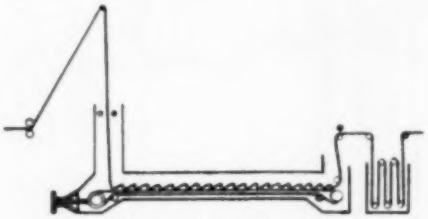

FIG. 9—Ganeval and St. Genis Machine

vessel with an entrance chimney, through which the material enters on to an endless band of stainless steel operating under the surface of the bleaching solution. The material is arranged on the endless band, on which rollers effect continuous squeezing, thus facilitating penetration and wetting of the material. Normally the total cycle of operations is so arranged that these treatments take place in one hour.

This machine is claimed to have the advantage over the American method of proceeding directly with the treatment of the material as it leaves the loom, and as the desizing may also be carried out continuously, regulation of this machine is stated to be less complicated than that of the American machines. It is claimed to lend itself to a variety of different materials to be treated; means of control are simple, and the quantity of material circulated is small. Its production is easily adjusted to small tonnages, and the flow of material, although still continuous, can be adapted to normal working hours.

(2) DUSGLER MACHINE—In this machine treatment is made in open width, which ensures uniformity of treatment. The ease with which the process is regulated makes it possible to pass immediately from one quality to another. The floor-space required is much reduced. The machine, shown diagrammatically in Fig. 10, comprises—(i) a padding machine for impregnation, (ii) a heating chamber with jets of steam on the two faces of the material, and (iii) a washing machine.

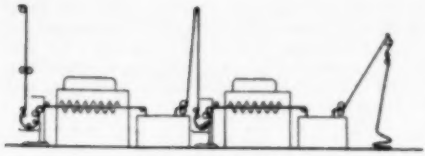

FIG. 10—Dungler Machine

HANDLING AND STORAGE OF HYDROGEN PEROXIDE

Methods of bulk storage and handling have been introduced for the large quantities of hydrogen peroxide employed in such fields as that of continuous cotton bleaching. Large containers have been developed, some of 10,000 gal. capacity. These tanks are usually of aluminium, but it is important that certain details of fabrication should be observed, and that a satisfactory chemical treatment of the aluminium be applied before the tanks are filled with hydrogen peroxide. In addition, the purity of the hydrogen peroxide plays a part. But, provided all these points are observed, experience has shown that there is no difficulty in handling any strength of solution of hydrogen peroxide. Suitable pumps and other auxiliary equipment necessary for bulk handling have also been developed. Hydrogen peroxide may be transported in road tankers.

SIMULTANEOUS BLEACHING AND DYEING

The cotton-bleaching technique discussed above is a good example of low process and labour costs, although large capital costs are involved for plant. Another such development of more immediate interest to the practical dyer, and one which does not involve alterations in existing plant, is the simultaneous bleaching and dyeing of cotton goods to obtain bright pastel shades.

The normal procedure for the production of clear light shades is to bleach the cotton in one operation, wash well with water, and then dye as a separate operation. Hydrogen peroxide is well suited for bleaching cotton under conditions normally employed in dyeing with direct dyes.

Before carrying out combined bleaching and dyeing, it is necessary to test the suitability of the dyes to be employed. This may be achieved by means of a boiling test to determine the resistance of the dye to change of hue or depth of colour. The test devised for this purpose comprises boiling under reflux a solution of dye in bleaching liquor containing hydrogen peroxide and sodium silicate. The composition of the solution should be the same as that to be employed in full-scale operation. As an example, the quantities required on the basis of dyeing to a 0·05% shade at 30 : 1 liquor ratio (assuming a 10-g. sample of cotton) are as follows—

Distilled water	300 ml.
Dye	0·005 g.
Sodium silicate	0·225 g.
Hydrogen peroxide (100-vol.) ...	1·5 ml.

These quantities represent 0·75 g. sodium silicate per litre and 0·5-vol. hydrogen peroxide. For convenience of measurement, stock solutions may be prepared when the boiling tests are to be carried out.

It has been found in general that the dye should withstand boiling for at least 30 min. without appreciable reduction in depth or change of hue if it is to be employed in the simultaneous bleaching and dyeing process. It was also found that the dyes which were graded as satisfactory by the boiling test can in general be employed for simultaneous bleaching and dyeing by only slight modifications of the methods recommended by the dye manufacturer.

The best method of application of particular dyes will be determined most satisfactorily under individual dyehouse conditions, but a method which has given satisfactory results with the majority of dyes examined is as follows—

Dye	Quantity to produce shade required
Glauber's salt	5–15% (on wt. of cotton)
Sodium silicate (78° Tw.) ...	12 oz./100 gal. liquor
Hydrogen peroxide (100-vol.)	3–5 lb./100 gal. liquor

The liquor : cloth ratio will be that normally employed for the dyeing operation. Dyeing is begun at approx. 40° C., and the solution raised to the boil in 15–20 min. and maintained at that temperature for 60 min. Sufficient soda ash is then added to the solution to raise the pH to 9·5–10·0, when the rate of decomposition of hydrogen peroxide is increased. Boiling is then continued for a further period (30–60 min.) in order to complete the bleaching process and decomposition of residual hydrogen peroxide.

Examples are available of direct dyes which have given consistent results using this method, although with some dyes the depths obtained will not match exactly those given in the pattern book using a given quantity of dye.

BLEACHING OF MIXTURES

The production of fabrics containing mixtures of fibres is of particular interest when the cost of many raw materials is high, and at the present time, for instance, wool is mixed with cheaper fibres in order to produce articles at economical prices. Mixtures and unions are produced both for cheapness and in order to confer desirable properties such as strength of material. On account of the large number of possible types of mixtures and unions it is not possible to deal with them in detail. It has, however, been found in practice that certain general principles may be employed when bleaching such mixtures.

The most common mixtures at the present time are those which contain two or more of the following — wool, cotton, viscose rayon, cellulose acetate rayon, nylon, and Ardil. The bleaching methods employed on such mixtures are governed by several factors, e.g.— (i) the fibre in the mixture most susceptible to attack by hydrogen peroxide, (ii) the tension to which the material is subjected during processing, (iii) the condition of the goods before bleaching (i.e. the necessity for removal of oil, size, etc.), (iv) the standard of bleach required.

It is usually found that mixtures containing wool (except mixtures with cellulose acetate) may be treated as for pure wool. Tests for chemical damage carried out on such unions have confirmed that wool-bleaching methods do not cause excessive damage to the secondary fibre. Mixtures of cotton and viscose are quite common and can be processed as for cotton only, if somewhat milder scouring conditions and reduction in temperature of the bleaching bath are ensured. Of the other fibres mentioned, care is required with cellulose acetate, as it is susceptible to alkalis and high temperatures, and it is our experience from laboratory trials that nylon is only slightly affected except under somewhat stringent conditions of bleaching with hydrogen peroxide. Ardil may be processed as for wool, although in mixtures with cotton or viscose rayon higher temperatures up to 175°F. can be employed; above this temperature a harsh effect is caused with alkaline liquors.

USE OF STANDARD DYEING EQUIPMENT

The work so far described in this paper has mostly concerned developments depending upon a change in the basic chemical principles or upon major changes in plant and method of handling the textile. During the past few years, however, many developments have taken place in the form of modifications to known bleaching solutions and application of known methods in different machines. One practical outcome is that under certain conditions types of dyeing machinery in regular use in the trade may be applied to bleaching with little or no adaptation. This, of course, includes the simultaneous bleaching and dyeing technique already described, and it has in addition been applied successfully to the bleaching of cellulosic fibres in the form of cloth, yarn, tapes, and ribbons in such types of dyeing machinery as winches and yarn-dyeing, pressure dyeing, and beam-dyeing machines. Beam and pressure dyeing machines permit bleaching in package form and simplify handling problems. Packages in which textiles have been bleached include beams, cheeses, cones, and cakes.

The efficiency of bleaching in such machines has been found in practice to permit substantial reductions in time of processing; in fact, satisfactory bleaching is often obtained in 1–3 hr. according to conditions and requirements, while by the older kier method the time of treatment for the bleaching stage was of the order of 5–6 hr. The solution employed in such machines for bleaching cotton is similar in composition to that employed in kier bleaching, e.g.—

Hydrogen peroxide (100-vol.)	...	3–5 lb.
Sodium silicate (78° Tw.)	...	3–8 lb.
Caustic soda	...	4–8 oz.

per 100 gal. solution. Liquor ratios of 5–10 : 1 are usually employed and the bleaching is carried out at the boil. Cotton bleached by the above method undergoes little chemical modification, and fluidity numbers of less than 3 have been obtained in works practice.

Owing to the short time of bleaching required in these machines, a quantity of hydrogen peroxide often remains undecomposed after completion of the bleaching operation. The most economical procedure is, therefore, to employ a stock tank, in which the solution is regenerated with hydrogen peroxide and stabiliser before each bleaching operation. In this way considerable savings may be made in steam and chemicals employed. Whereas it has been normal practice to run the exhausted bleaching solution to waste after each operation, a stock solution maintained as described may be used for several weeks to advantage.

* * *

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COMMUNICATION

The Desorption of a Direct Cotton Dye from Cellulosic Fibres

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A study has been made of the desorption of purified Chlorazol Sky Blue FF from dyed bleached cotton and from dyed viscose rayon into water and into solutions of sodium chloride. With cotton a dyeing hysteresis is observed. Unexpected features of the desorption into very dilute solutions of sodium chloride have been explained by assuming that the cotton may contain a small proportion of basic groups.

Introduction

The dyeing of natural and regenerated cellulosic fibres by direct cotton dyes is now accepted as essentially a reversible process. It is unnecessary at this stage to review the large amount of experimental work which has demonstrated the ultimate establishment of reproducible equilibria in carefully prepared fibre–dyebath systems, and which has defined the influence of each of the common variables in the total dyeing process upon the position of this equilibrium [1]. Because this equilibrium is reached, the uptake of dye by the fibre must be the resultant of two opposing processes, adsorption and desorption. While the fact of desorption has been accepted, quantitative investigation and theoretical speculation have tended to place the greatest emphasis upon the adsorption process. Desorption, which limits the proportion of dye that can be transferred from bath to fibre, and which is directly connected with lack of fastness to wet treatments, limits to some extent also the technical value of the dye.

Nevertheless, quantitative information about the desorption process should be useful for the following reasons—

(i) The relative ease of desorption of a dye from a fibre should help to define its fastness towards wet treatments if there is no accompanying modification of the dye or the fibre-substance.

(ii) Since the possibility of correcting initial unlevelness of dyeings by prolonging the dyeing time depends essentially upon the ability of the dye to leave the fibre, a quantitative assessment of the desorption characteristics of the dye is an essential step to a definition of its levelling power.

(iii) The mechanism by which direct dyes combine with cellulose to provide maximum fixation has been assumed to consist in the formation of at least two hydrogen bonds between each dye molecule and the molecule of the substrate. In a dyeing, some dye molecules may be less firmly attached, e.g. by means of one hydrogen bond only, and it may be possible ultimately to demonstrate this by a study of the kinetics of desorption.

(iv) While it is possible, by careful reproduction of the experimental conditions, to reproduce accurately the equilibrium partition of dye between the solution and the fibre-substance, neither the dye, nor especially the fibre, can be accepted as completely stable. If the experiment is carried much beyond the time required to obtain an apparent equilibrium, further changes in the partition may result. Experiments in which desorption is made to predominate must always be preceded by those in which the fibre has been dyed for a period sufficient to ensure even distribution of dye through the diameter of the fibre. As the time of contact with the hot dyebath is increased, secondary changes, e.g. in aggregation of dye within the fibre or in the fibre structure itself, may take place. These changes may show up more clearly in desorption experiments.

(v) Factors which have been shown to influence strongly the kinetics of dyeing, such as the differences in electrical potential which are established at the surface of separation between fibre and solution, have, so far, been considered more in relation to their effect upon the migration of dye into the fibre than upon the reverse movement of dye from fibre to dyebath.

With these considerations in mind, a preliminary examination of the desorption of a typical direct dye from dyeings on cotton and on viscose rayon was begun. The cotton was in the form of thoroughly scoured and bleached 30/2 soft-twisted Egyptian yarn (English Sewing Cotton Co.); the viscose rayon a yarn of comparable structure, 30/2 spun Fibro, 1·5 fibre denier (Courtaulds). The dye was a purified specimen of Chlorazol Sky Blue FF (ICI; *C.I.* 518). The apparatus and procedure were essentially those described by Neale and Stringfellow [2] for accurate dyeing experiments, with some modifications to ensure that entrained dye liquor was removed as completely as possible from the sample of yarn at the end of the desorption.

Summary of Results

(1) Desorption of Chlorazol Sky Blue FF from equilibrium-dyed cotton or viscose rayon represents a qualitative reversal of the absorption process in that removal of dye from the fibre to the external solution proceeds smoothly, and at a constantly diminishing rate, until equilibrium is reached.

(2) Under comparable conditions, desorption takes place at a greater rate than adsorption. This applies both to bleached cotton and to viscose rayon.

(3) The rate of desorption from cotton increases with increase in the concentration of sodium chloride in the external bath. With all other conditions maintained constant, it increases with increase in the temperature.

(4) The concentration of dye in the fibre, when it is in equilibrium with a given concentration of dye in the solution, is greater if the equilibrium has been approached by way of desorption than by way of adsorption. A dyeing hysteresis thus exists, and it is found to increase in magnitude as the temperature falls.

(5) When the cellulose is in the form of bleached cotton, the dye remaining in the fibre at equilibrium is greater if desorption has taken place into pure water than into salt solutions over the range 5–20 g. per litre. The dye remaining in the fibre at equilibrium is least for a salt concentration of 0·5 g. per litre, rising slowly thereafter as the concentration increases.

(6) With viscose rayon staple fibre, on the other hand, the dye remaining in the fibre at equilibrium is least when desorption takes place into pure water, and rises steadily as the salt concentration in the external solution increases.

(7) To explain the results set out in (5) and (6) above, it is suggested that, even in carefully scoured and bleached cotton, there may be a small residual proportion of free basic groups.

Experimental

MATERIALS

COTTON—In most of this work, cotton cellulose has been used, the original intention being to avoid the disturbing effects of the high carboxyl content of regenerated forms of cellulose upon dye absorption. Yarn was chosen in preference to cloth, since it is easier to obtain pure and to manipulate in the dyeing experiments. A good-quality 30/2 bleached, combed, soft-twisted Egyptian yarn (English Sewing Cotton Co. Ltd.), taken from a single 1-lb. cone, was used throughout the work. It was further treated to remove soap or other possible softening agents by heating for 1 hr. at 60°c. in dilute ammonia solution (10 ml. ammonia (sp. gr. 0·88) per litre), washing five times separately in hot distilled water, drying at 110°c., and conditioning as described below. Carboxyl content (methylene blue method [3])*—as conditioned, 0·66 millimole per 100 g. dry cotton; after souring in 0·1 N. hydrochloric acid, washing until free from acid, drying, and conditioning, 0·66 millimole per 100 g.

VISCOSE RAYON—30/2 Soft-twisted, spun Fibro yarn (Courtaulds), of filament denier 1·5, was treated in dilute ammonia solution, washed, and dried as for cotton. Carboxyl content*—dried and conditioned, 2·63 millimoles per 100 g. dry fibre; soured and washed as above, 2·52 millimoles per 100 g.

DYE—A commercial sample of Chlorazol Sky Blue FF (ICI) was purified according to the method of Mills and Robinson [4] by repeated precipitation from a strong aqueous solution through the addition of solid sodium acetate. During the precipitation, a small proportion of ethyl alcohol was added to ensure that the dye came down in a form that could be readily filtered. When no chloride or sulphate ions could be detected in the filtrate, the dye was collected, dried as rapidly as possible at 110°c., extracted in a Soxhlet with dry ethyl alcohol until all the acetate had been removed from the dye (cacodyl test), and dried in thin layers at 110°c. for two days and then *in vacuo* over phosphorus pentoxide for one week. It was preserved over phosphorus pentoxide. The purity was checked colorimetrically by purifying until the optical density in a standard solution remained unchanged, and also by comparison with other pure specimens.

SODIUM CHLORIDE—AnalaR reagent was dried to constant weight, and kept in a desiccator over phosphorus pentoxide.

PYRIDINE—Pyridine for extraction of dye from dyed samples [5] was redistilled, the fraction boiling at 112–115°c. being retained and diluted with distilled water (20 g. pyridine per litre).

APPARATUS

DYEING—The apparatus was essentially the same in construction as that used by Neale [2]. The dyeing vessel was a Pyrex glass tube, of approx. 130 ml. capacity, fitted, by means of a ground glass joint at the mouth, with a short Liebig condenser also in Pyrex. The dyeing tubes were immersed in sets of six in an oil-filled thermostat, heated electrically and controlled by a mercury–toluene regulator operating through a thermal switch. The oil was circulated by means of a propeller, and was maintained normally at 98·5 ± 0·1°c. for a dyeing temperature of 95°c. The specimen of yarn was suspended in the dyebath at the lower end of a stout silver wire which passed through the condenser and hooked to a reciprocating bar driven through a gear by a synchronous electric motor. The specimen was thus caused to oscillate vertically 130 times per minute through a distance of 7 cm. Following other workers, it was found that regularity of agitation of the specimen in the dyebath was essential if consistent results were to be obtained.

ABSORPTIOMETER—The standard model of the Hilger Spekker photoelectric absorptiometer was used throughout the work.

CENTRIFUGE—As will be discussed later, consistent results for the amount of dye left on the fibre, after dyeing or desorption experiments had been carried out, could be obtained only if special precautions were taken to remove dye liquor entrained in the yarn without disturbing the dyed dye. This operation was performed in a small bench centrifuge with a specially designed tube, made from a common type of glass centrifuge tube, 11 cm. long and 1·5 cm. in diameter at the mouth, the upper half being cylindrical, and the lower tapering almost to a point. The pointed end was opened, and the tube constricted to about half the full diameter about half-way along its length. A small funnel was made, 1 cm. in diameter at the mouth with a stem of thick-walled capillary 2·5 cm. long and of 1 mm. bore. It passed, stem downwards, through a rubber washer held tightly in the constriction of the main tube, so that the latter was separated into two compartments communicating only by the capillary of the stem. It was thus possible, with the tube upright in a bucket of the centrifuge, to place the dyed specimen in the funnel, cover it with a small, known volume of water, and so to wash it as the centrifuge was operated.

PROCEDURE

Only by most careful adherence to the following procedure was it possible to obtain results which were sufficiently accurate and consistent to establish the small differences in dye content

* We are indebted to Dr. T. P. Nevell of the British Cotton Industry Research Association for these determinations.

resulting after adsorption and desorption respectively for certain concentrations of dye and electrolyte in the dyebath.

PREPARATION OF YARNS—Undyed yarn for carrying out adsorption experiments and dyed yarn for carrying out parallel desorption experiments were required. In each case, 34-cm. lengths of the yarn (weighing just over 10 mg.) were wrapped into small skeins 6 cm. long, tied in loosely with a few centimetres of free yarn beyond the knot. Skeins for desorption experiments were first dyed, 60 at a time, at 95°c. for 24 hr. in a bath containing 3·0 g. pure Chlorazol Sky Blue FF and 5·0 g. sodium chloride per litre. After removal from the dyebath, the batch of dyed skeins was washed in a sintered glass filter-funnel with 200 ml. cold water and hydroextracted at 2000 r.p.m. for 1 min. The skeins were conditioned at 64% R.H. for 2 weeks and each was weighed on an accurate torsion balance, the loose ends being trimmed until the final weight was exactly 10 mg. cotton, allowance for the dye present being made after it had been suitably determined on a number of skeins from the batch. The rest were then replaced in the conditioning atmosphere until required. The skeins for adsorption experiments were treated in exactly the same way, except that, instead of the dyeing operation, they were kept in distilled water for 24 hr. at 95°c. to ensure that the previous history of cotton used for adsorption was as close as possible to that used for desorption. The corresponding sets of Fibro yarn were treated in exactly the same fashion as the cotton.

DYEING OR DESORPTION—This was carried out for the required time and with the desired bath composition on single skeins in the apparatus already described. The volume of the bath was standardised for most of the experiments at 100 ml.

WASHING-OFF—Some time was spent in finding a satisfactory procedure. In earlier experiments, largely with sheet cellulose, Neale [2] had removed loose dye liquor by blotting the specimens between sheets of filter-paper, and even with this form of cellulose an increasing scatter in the experimental values had been found as the concentration of dye in the bath was increased, pointing to interference from dye in the imbibed liquor not fixed by the cellulose. With yarn, which contains a proportionately large total volume of interfibre capillaries, the interference was much greater. On the other hand, with any prolonged rinsing treatment, the danger of removing dyed dye increased. Ultimately, the most satisfactory results were obtained as follows. The silver wire with the skein attached was removed from the dyebath at the end of the desired time, and the specimen agitated in cold distilled water for 1 sec. largely to cool it and to arrest further dyeing. The skein was at once transferred to the funnel of the centrifuge tube, covered with 4 ml. of distilled water, and centrifuged at 2000 r.p.m. for 1 min.

DETERMINATION OF DYE CONTENT—Immediately after centrifuging, the specimen was transferred to a stoppered bottle, covered with a measured volume of 20% aqueous pyridine, and allowed to stand at room temperature for 2 days. The concentration was determined in the usual way in the Spekker, using a 4-cm. cell and the appropriate filters. The instrument had previously been calibrated with a series of standard solutions of the pure dye in 20% aqueous pyridine. The volume of dilute pyridine used for extracting the dyed specimen was adjusted according to the amount of dye on the yarn, so that under the stated conditions the Spekker readings were near the middle of the scale, where the sensitivity of the instrument, which possesses a logarithmic scale, is still sufficiently great.

Before the procedure was finally adopted, two possible sources of error were investigated—

(i) It was found that no significant difference could be observed in the dye content of specimens dyed under the same conditions when the temperature of the rinsing water was changed from 0° to 18°c. If rinsing were done at 40°c. consistently lower values for dye content were obtained, and it was concluded that some removal of dyed dye was taking place. 18°c. was therefore taken as the rinsing temperature.

(ii) Neale and Fishwick [6] have reported marked decomposition of Chlorazol Sky Blue FF in a bath maintained at 90°c. overnight with cotton and salt present. The following experiments were therefore made—A solution containing 0·5 g. dye and 5·0 g. sodium chloride per litre was divided into two. One portion was heated for 1 week at 100°c., the other maintained in the dark at room temperature. Each was then diluted by the same amount with aqueous pyridine and the two were compared colorimetrically. No difference could be found. A similar pair of solutions was prepared, and dyeings were carried out under identical conditions with each of them. No difference attributable to heating one of the solutions could be detected in the dye content of the resultant dyeings. As a further test, a number of small cotton skeins were dyed together in a bath of volume large enough to ensure that the dye concentration was not appreciably diminished by adsorption on the fibre. The experiment continued over one week, and after a time sufficient for normal equilibrium adsorption to be established, samples were removed at intervals and the dye contents determined. No change in dye content with time could be detected. We are therefore unable to account for the results of Neale and Fishwick, but are satisfied that the risk of decomposition need not be taken into account in the present experiments.

Experimental Results

COMPARISON OF ADSORPTION AND DESORPTION

In Fig. 1 is shown the course of dye transfer between either (i) bleached cotton or (ii) equilibrium-dyed cotton containing 0·96 g. of dye per 100 g. cellulose, and a dyebath containing initially 0·1 g. of pure dye and 5·0 g. sodium chloride per litre at 95°c. It will be seen that in the initial stages of desorption dye is transferred more rapidly than in the initial stages of adsorption, and that the equilibrium dye content of the cotton is greater in the desorption experiments. The difference in the respective equilibrium values is far greater

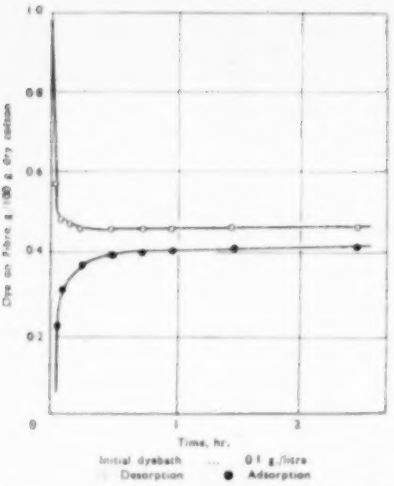

FIG. 1.—Adsorption and Desorption of Pure Chlorazol Sky Blue FF for Cotton at 95°C.

than can be accounted for by the difference in the corresponding final dye concentrations in the bath (viz. adsorption 0·0990 g. and desorption 0·1005 g. of dye per litre) when allowance is made for the dye taken from, or given up to, the bath. The same behaviour was found in similar sets of experiments in which the initial dye content of the bath ranged from 0·05 to 1·00 g./litre and all the other conditions remained the same. The results for the two extreme dyebath concentrations are given in Table I, where the adsorption A_t at any time t is presented as a fraction of the equilibrium adsorption A_i. The desorption is similarly expressed as D_t/D_i. This gives a more satisfactory basis for comparison than the actual values for adsorption or desorption, since, as the dye concentration of the bath increases, the absorbing fibre must take up relatively more, and the desorbing fibre lose relatively less, to reach equilibrium.

TABLE I

(Pure dye on cotton at 95°C. NaCl 5·0 g./litre)

Dye initially in bath, g./litre ...	0·05		1·0	
Time (min.)	A_t/A_i	D_t/D_i	A_t/A_i	D_t/D_i
2 ...	0·39	0·65	0·68	0·79
5 ...	0·68	0·84	0·85	0·90
10 ...	0·87	0·96	0·93	0·97
30 ...	0·98	1·00	0·98	1·00
60 ...	1·00	1·00	1·00	1·00
120 ...	1·00	1·00	1·00	1·00

These results, for desorption only, are further represented in Fig. 2 alongside results of experiments where desorption is taking place into a bath consisting initially of distilled water only, and also into a bath containing initially 5·0 g. sodium chloride per litre but no dye. It is seen that desorption is slowest into water alone, is somewhat more rapid when salt is present, and, in relation to

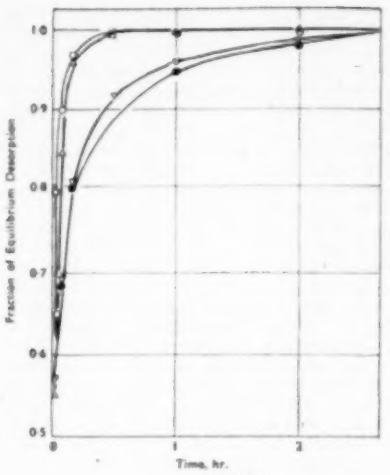

FIG. 2.—Desorption of Pure Chlorazol Sky Blue FF from Cotton into Different Solutions at 95°C.

the amount of dye transferred, is more rapid still when some dye, even at low concentration, is initially present in the solution.

DESORPTION FROM DYED VISCOSE RAYON

Similar experiments to those described above were carried out, at one dye concentration only, with the spun Fibro yarn. The changes of A_t/A_i and D_t/D_i with time are shown in Fig. 3, alongside comparable results for cotton. The initial dye content of the cotton yarn used in the desorption experiments was 0·84 g. per 100 g. dry fibre; but, to take into account the much higher equilibrium exhaustion of the Fibro, the dye content of the yarn used for desorption was 2·2 g. per 100 g. of dry fibre. As already observed for cotton, the relative rate of desorption is greater than that of adsorption. The results also agree with the observations of Boulton and Morton [7], who showed that, while cotton is dyed more rapidly in the initial stages than viscose rayon, equilibrium is finally attained more rapidly by the latter. The results suggest that this is also true for the desorption process, cotton desorbing more rapidly at the beginning of the process and viscose rayon more rapidly as equilibrium is approached.

EFFECT OF TEMPERATURE ON DESORPTION

This effect was not studied in great detail, but the curves in Fig. 4 for desorption from cotton containing initially 0·97 g. of dye per 100 g. into a solution containing 5·0 g. of sodium chloride per litre but with no dye present at the beginning show that the rate of desorption, as might be expected,

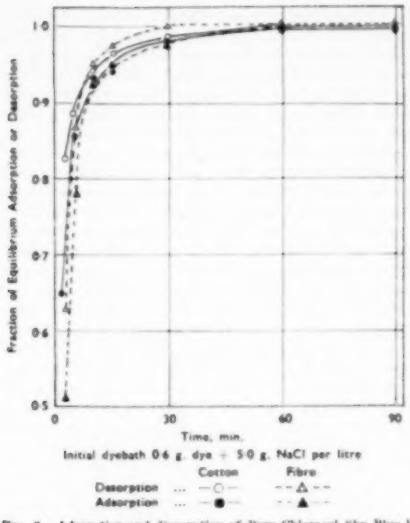

FIG. 3—Adsorption and Desorption of Pure Chlorazol Sky Blue FF for Cotton and Viscose Rayon at 95°C.

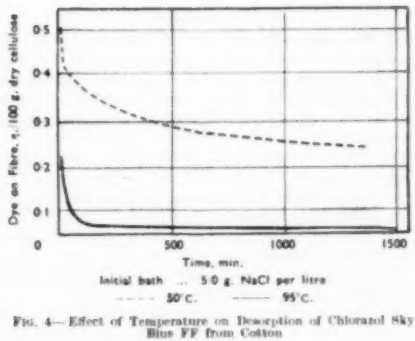

FIG. 4—Effect of Temperature on Desorption of Chlorazol Sky Blue FF from Cotton

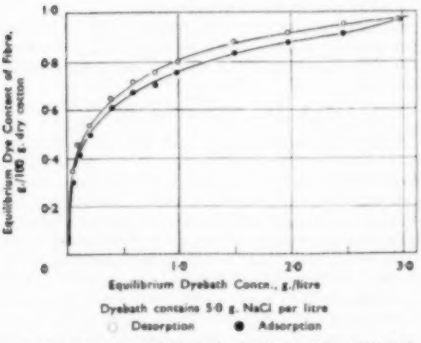

FIG. 5—Adsorption and Desorption Isotherms of Pure Chlorazol Sky Blue FF for Cotton at 95°C.

increases as the temperaure is raised from 50° to 95°C.

DYEING HYSTERESIS FOR COTTON

In Fig. 5 are shown adsorption and desorption isotherms for cotton at 95°C. in the presence of 5·0 g. of sodium chloride per litre in the dyebath. It will be seen, in agreement with the published data of other workers, that the dye content of the fibre at equilibrium, whether this has been reached by a process of adsorption or a process of desorption, increases with increasing concentration of dye in the bath. Except for the highest dyebath concentration (3 g. of pure dye per litre), where, for reasons already discussed, the accuracy of the determinations is likely to be less, the desorption equilibrium content of the fibre is consistently higher than the adsorption value, the difference over the whole range of dyebath concentration being almost constant at 0·04–0·05 g. of dye per 100 g. of cotton. It has been shown that the adsorption of certain direct dyes on cellulose is of the Freundlich type, and Standing and co-workers [8] suggest that the slope of the curve of the logarithm of the dye concentration in the fibre against the logarithm of the dye concentration in the bath should be $1/(z + 1)$, where z is the valency of the dye ion. For Chlorazol Sky Blue FF ($z = 4$) this value is therefore 0·2. The logarithmic curves for both the adsorption and desorption isotherms given in Fig. 5 are shown in Fig. 6. It will be seen that a considerable proportion of the middle range of each curve is linear, the slope of the adsorption isotherm being 0·28 and of the desorption isotherm 0·25. The agreement with the theoretical value is thus reasonably good, being rather better for the desorption than for the adsorption isotherm.

For cotton there is thus an unmistakable dyeing hysteresis. The values given appear to be true equilibrium values both for adsorption and for desorption, since further experiments showed that they remained constant over periods ranging from 2 hr. to 3 days. The difference between corresponding equilibrium values for adsorption and desorption cannot be ascribed to experimental error, the maximum value of which was of the order of 1%.

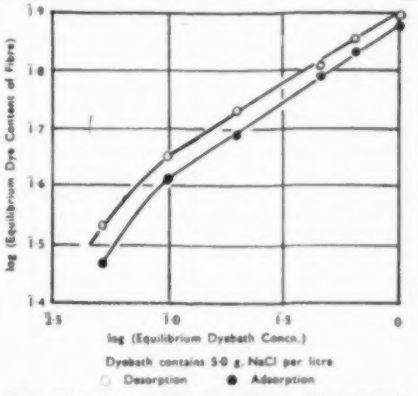

FIG. 6—Adsorption and Desorption Isotherms of Pure Chlorazol Sky Blue FF for Cotton at 95°C.

while the difference was approx. 6·2% of the adsorption equilibrium value.

As already stated, the influence of temperature was not studied in detail, but the results given in Table II for cotton at one dyebath concentration suggest that, in agreement with the results of other workers, the equilibrium content of the dye attained by adsorption diminishes with rising temperature. This also seems to be true of the desorption equilibrium value, and the difference between the two, the hysteresis value, also decreases as the temperature rises.

TABLE II

Equilibrium Dye Contents on Cotton

(g./100 g.)

(Cotton, 0·03 g.; pure dye + 5·0 g. NaCl per litre)

Temperature, °C.	50	70	95
Adsorption (*A*)	0·766	0·445	0·215
Desorption (*D*)	0·826	0·498	0·257
Hysteresis (*D* — *A*)	0·060	0·053	0·042

From a limited number of adsorption and desorption experiments with viscose rayon the equilibrium hysteresis at 95°C. in a bath initially containing 0·6 g. of pure dye and 5·0 g. of sodium chloride per litre was found to be 0·145 g. of dye per 100 g. of fibre, and is thus much higher than the value for cotton (0·044 g.) under the same experimental conditions.

EFFECT OF SODIUM CHLORIDE CONCENTRATION ON DESORPTION

One series of experiments was made in which desorption took place into baths containing no dye initially but with different concentrations of sodium chloride. The results are shown in Fig. 7, where the dye content of the fibre on desorption of equilibrium-dyed fibre to equilibrium with the salt bath is plotted against the sodium chloride concentration. With cotton, the unexpected result was obtained that desorption into distilled water is less complete than into sodium chloride solutions at any of the concentrations studied, the desorption equilibrium value falling sharply on the addition of 0·5 g. of salt per litre, and then rising slowly as the salt concentration increased, but never reaching the value attained with water alone. With Fibro, on the other hand, desorption was more complete into pure water, and diminished steadily as the concentration of sodium chloride increased. As set out more fully in the discussion, it is suggested that the distinctive behaviour of cotton may be due to a relatively small number of basic groups in the fibre, a conclusion reached by other workers, so that the first small additions of salt have the same effect upon dye adsorption as they would have on the adsorption of an acid dye by a protein fibre. To check this, desorption experiments were carried out with cotton and with viscose rayon which had been dyed with pure Coomassie Red PG and pure Coomassie Brilliant Blue R, both acid dyes that have some affinity for cellulose. The results did not agree with those obtained with Chlorazol Sky Blue FF, desorption from both cotton and viscose rayon being greatest into pure water and diminishing as the salt concentration increased. It was not possible to carry this investigation very far, and in view of certain additional difficulties in experiments with these dyes, it would be advisable to regard the results as inconclusive until a more extended examination can be given.

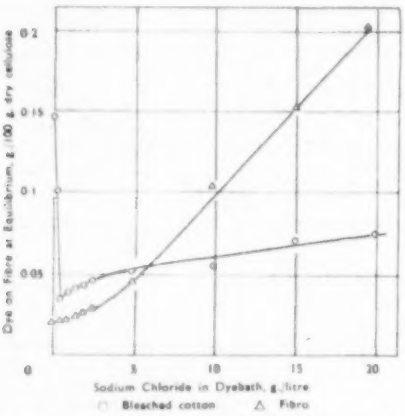

FIG. 7.—Desorption of Chlorazol Sky Blue FF from Cotton and Viscose Rayon at 95°C. into Salt Solutions of Different Concentrations

Discussion

The evidence set out earlier in this paper suggests that the dyeing of bleached cotton and of viscose rayon is not strictly reversible under the conditions employed (Fig. 5). Observations which are not at once predicted by present dyeing theory include—

(1) The dye content of the fibre in equilibrium with a dyebath of given concentration is greater when equilibrium has been reached by desorption than when it has been reached by adsorption.

(2) The relative speed of desorption is greater than that of adsorption.

(3) Desorption is accelerated by the presence of electrolyte in the dyebath.

(4) Desorption from equilibrium-dyed cotton is least into pure water, being greatest in very dilute solutions of sodium chloride, and diminishing as the salt concentration is increased.

(5) The slope of the desorption isotherm agrees more closely with a theoretically predicted value than does the slope of the corresponding adsorption isotherm.

LACK OF REVERSIBILITY

Evidence brought forward for the reversibility of direct cotton dye adsorption is of two kinds. Boulton, Delph, Fothergill, and Morton [9], Neale and co-workers [1], Gnehm and Kauffler [10], and Weltzien and Schultze [11], using a variety of direct dyes, report that the dye can ultimately be removed completely from the cellulose by washing with distilled water. Other evidence is based on the reproducibility of the equilibrium condition when approached from either side (Neale [1]). The two kinds of criteria are not quite equivalent in significance, since, unless desorption equilibrium is

allowed sufficient time for its establishment, intermediate apparent equilibria will be affected by differences in the strength of chemical attachment between dye and fibre among the total number of dye molecules in the system. Gnehm and Kauffler [10] observed as early as 1902 that a slight residue of the dye was more difficult to remove from the fibre than the rest. The unusual fastness to washing of a number of dyes, including Benzopurpurin 4B, when dyed on cotton in pale shades is well known, and this has been further emphasised by Peters and Vickerstaff [12], who have suggested that it may be caused by the presence of a small proportion of basic groups in the fibre, a suggestion which seems to be confirmed in detail by some of the present results (Fig. 7).

A suggestion has been made in private discussion by Dr. T. H. Morton that the hysteresis now observed is specifically connected, in the following manner, with the experimental conditions employed— During the preparation of dyeings for desorption experiments, the cotton was dyed for 24 hr. Equilibrium adsorption is approached very closely in less than 2 hr., and although no further structural change in the fibre is indicated by changes in the adsorption during the next 22 hr., it is suggested that the cellulose fibre structure does in fact change in such a way that some of the adsorbed dye molecules become more difficult to desorb. This suggestion is now being investigated.

There are, nevertheless, factors which have been shown, or have been assumed, to affect both the rate and the extent of adsorption, e.g. differences in electrical potential between dyebath and fibre substance at the common interface, the effects of which on the reverse process of desorption seem to have received scarcely any consideration. When cellulosic fibres are immersed in a dye solution containing salt, a high concentration of dye ions is very rapidly established in the peripheral layer, from which a steady diffusion takes place into the body of the fibre. The implicit assumption of Neale was that this surface layer had a concentration proportional to the external dyebath concentration, the latter and, in consequence, the former being maintained constant by working with a bath of so great a volume that there was no appreciable change in concentration during the course of dyeing. Crank [13] has shown, however, that mathematical solutions for rate and extent of diffusion into the interior of the fibre, worked out originally for the infinite bath, tend to be valid when the bath is restricted in volume and loses a significant proportion of the initial dye present by adsorption on the fibre. Apart from discontinuities in the fibre structure which are known to exist (skins and cuticular processes), it is not difficult to imagine how the dye superficially adsorbed could exercise a progressive control on the further uptake of dye by the fibre, tending to delay it until some of the dye first adsorbed had diffused further into the centre. This could lead to the presence of an outer zone in the fibre in which, after the initiation of dyeing, the concentration would remain more or less constant in value. Crank inclines to the idea of a compensatory electrical mechanism, depending upon the repulsion of entering dye anions by those already fixed upon the fibre. A concentration barrier of the sodium gegenions of the dye in the outer zone would act in the same manner. It is also established that the diffusion coefficient increases with increasing concentration of the dye at any particular locality in the fibre, so that dye ions would require a relatively small difference of concentration to get them through the region of high concentration. From this view, the "initial concentration" as far as the diffusion within the fibre is concerned can be imagined as remaining constant, while the dye uptake during the later stages of adsorption is determined by the diffusion process. There are certain difficulties connected with this view when the effects of increasing concentrations of sodium chloride in the dyebath are considered, for these should tend to nullify the restrictive effects of both potential and concentration barriers on the primary uptake of dye into the peripheral region, and therefore the controlling influence of these factors should be diminished.

So far, no suggestions have been made which are based upon steps in the process of desorption when a fibre dyed to equilibrium is placed in water, in a dilute salt solution, or in a dyebath of concentration lower than that which corresponds to the equilibrium adsorption value for the dyeing. The simplest assumption would be that the dye is uniformly distributed through the fibre during the preparation of the dyeing, though there seems to be no direct evidence that equilibrium dyeings are in fact completely uniform. As the greatest concentration gradient in the system will now be at the interface between the dyed fibre and the surrounding bath, diffusion is to be expected first from the outer layers of the fibre. The outer layers will thus be the site of a diminishing concentration of dye, and further dye will diffuse into them from the inner regions. Thus, although the dye distribution curve, in passing from the inside to the outside of the fibre, will have a general slope for desorption which is in the inverse sense from that which obtained for the adsorption, it is difficult to see that there can be any correspondence between the point-to-point details. Since the diffusion coefficient diminishes as the concentration falls, it would seem that dye molecules from the inside of the fibre will meet with increasing difficulty in passing through the outer layers as these become denuded of dye: i.e. such molecules incur an increasing risk of re-adsorption on the cellulose, although combating this will be an increasing steepness of the concentration gradient. However, there is nothing in the picture as it stands, lacking quantitative data on the dye distribution within the fibre, which would predict the existence of a hysteresis between adsorption and desorption.

The electrical potential barrier has been generally invoked to explain the restricted adsorption of dye in the absence of appreciable concentrations of added electrolytes. It is important to enquire how it may affect the desorption process. If the barrier is considered as a single step from a substantially equipotential dye solution to an equipotential fibre, the observed potential difference would be

expected to assist the escape of dye ions from the fibre to the bath. As part of the potential difference is due to the dye ions adsorbed on the surface, this assistance would be expected to diminish as desorption proceeds. Similarly, the difference of concentration of sodium ions between a dyed fibre and the external bath (Donnan effect) would be expected also to assist desorption. These two effects would explain the relatively greater speed of desorption, but not the observed hysteresis. But the presence of added electrolyte, which diminishes both effects, increases the rate of desorption instead of diminishing it. As Entwistle [14] points out, the conception of the single potential step, located only at the surface of the fibre, may be an unjustifiable simplification. If, through local differences in the orientation of the fibre molecules at the surface, there is a corresponding local restriction in the movement of ions so that a local concentration of dye at this region is set up, whether it be fed from the dyebath during adsorption or from the inside of the fibre during desorption, a potential or a concentration "wall" rather than a "step" may be formed. It would then be possible to explain the effect of added electrolyte in increasing the rate and extent of desorption.

SPECIAL BEHAVIOUR OF BLEACHED COTTON

From Fig. 7 it will be seen that desorption is least in distilled water. A very small concentration of sodium chloride causes a marked increase, and further additions a progressive small decrease in desorption. This is to be contrasted with viscose rayon, in which desorption is greatest into plain water, and is appreciably diminished as salt is added. The carboxyl content of the rayon is four times as great as that of cotton. The effect of salt in diminishing the desorption of the viscose rayon may, following Neale, be ascribed to a diminution of the influence of electrical repulsion due to the surface charge on the fibre, as well as to a decrease in the escaping tendency of the sodium ions attached to the adsorbed dye molecules.

In bleached cotton, the initial decrease in the amount of dye present in the fibre after desorption, brought about by the addition of salt up to 0·5 g. per litre of dyebath, may, following Peters and Vickerstaff, be caused by the presence of a small proportion of basic groups. If these basic groups are few, and are therefore likely to be widely separated in the fibre, they should be more or less independent of the small number of carboxyl groups also present. On dyeing, the basic groups will become saturated with dye anions, and, on desorption, dye will be retained by them, probably up to the stage when most of the rest has been desorbed. The presence of chloride or other competing anion in the desorbing bath will, as with the acid dyes on basic fibres (proteins or nylon), tend to displace the dye from its combination with the basic groups and assist in the final stages of removal. Because of the small number of basic groups in the cotton, only a correspondingly small concentration of chloride ions will be necessary to accomplish this. Further additions of salt will suppress desorption, just as they do with viscose rayon, although, because there are fewer carboxyl groups in cotton than in viscose rayon, the relative effect will be less. The results given in Fig. 7 show that the viscose rayon, after a standard desorption procedure, contains relatively less dye than cotton, although originally more dye was present, thus supporting the above suggestion.

It is disappointing that a direct confirmation of the presence of basic groups or an estimate of their proportions could not be confirmed by measurements of the adsorption of the selected acid dyes. Subsequent work on the transfer of acid dyes to cellulose under printing conditions has shown a variety of transfer behaviours among different acid dyes, and other methods of approaching the problem will probably be necessary. The only definite conclusion which seems to be reached is that, even at a salt concentration which produces maximum desorption from cotton, some dye is strongly retained in the fibre by influences other than the postulated electrovalencies.

* * *

We desire to express our gratitude to the British Cotton and Wool Dyers' Association Ltd. for a grant which enabled one of us (J.H.E.J.) to take part in this work.

DEPARTMENT OF TEXTILE CHEMISTRY
COLLEGE OF TECHNOLOGY
UNIVERSITY OF MANCHESTER

(Received 20th May 1952)

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ERRATA

The Continuous Dyeing of Vat Dyes (the Standfast Molten Metal Technique). J. Ardron, M. R. Fox, and R. W. Speke. (J.S.D.C., **68**, 250 (July 1952))— The caption to Fig. 2 should read only— **Leuco Dyeing.**

Final Report on the Census of Production for 1948 (J.S.D.C., **68**, 309 (Aug. 1952))— The year was wrongly given as 1945.

Notes

Meetings of Council and Committees
August

Council— No meeting
Publications— No meeting
Colour Index Editorial Panel— 28th
Mercer Lecture— 22nd

Deaths

We regret to report the loss by death of Mr. O. Gunnell and Mr. G. L. Usher.

Reports on the Dyeing Properties of Dyes

The Reports of the Society's Committees on the Dyeing Properties of Direct Cotton, Vat, and Wool Dyes are now available in the form of a single combined volume. In a Foreword, Mr. F. Scholefield (*Immediate Past President*) says that the book "enables the inquirer to assess by means of simple tests those properties of any direct cotton, acid wool, or vat dye which are of importance in their respective dyeing processes." Copies may be purchased from the Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire, at 5s. 0d. each post-free.

The Chemical Society
Corday–Morgan Medal and Prize

This award, consisting of a silver medal and a monetary prize of 150 guineas, is made annually to the chemist of either sex and of British nationality who, in the judgment of the Council of the Chemical Society, has published during the year in question the most meritorious contribution to experimental chemistry, and who has not, at the date of publication, attained the age of thirty-six years.

Copies of the rules governing the award may be obtained from the General Secretary, Chemical Society, Burlington House, Piccadilly, London W.1. Applications or recommendations in respect of the award for the year 1951 must be received not later than 31st December 1952, and applications for the award for 1952 are due before the end of 1953. The first award was made during 1951 in respect of work published in 1949.

Polycyclic Hydrocarbons in Dye Synthesis

In a lecture to the Society of Chemical Industry in Manchester, Dr. T. Holbro said that the use in dye synthesis of polycyclic aromatic hydrocarbons, now available in large quantities, could have a beneficent effect on the properties of the dyes. The most interesting results were obtained with the tetracyclic compounds chrysene, fluoranthene, and pyrene, which could be used in the anthraquinone vat dye field.

German Patent Specifications

German patent specifications, receipt of which was interrupted by the outbreak of war in 1939, are again being received by the Leeds Library of Commerce and Technology. They may be obtained on loan by any library or industrial organisation in the United Kingdom by sending a written request to the City Librarian, Library of Commerce and Technology, Municipal Buildings, Leeds 1.

It is of interest to record that this library holds also a complete set of British Patents, dating from 1617, and United States Patents from 1893.

C O C

Production of Lakes and Toners in the U.S.A. in 1950 and 1951

The U.S. Tariff Commission reports that total production of lakes and toners was (all figures being millions) 47·4 lb. in 1950 and 46 lb. in 1951; sales were 43·7 lb. worth $54·4 in 1950 and 38·9 lb. worth $51 in 1951. Production of lakes or dyes for lakes was 10·1 lb. in 1950 and 9·9 lb. in 1951, of which Patent Blue (*C.I.* 671) accounted for 2·4 lb. Sales of lakes were 9·1 lb. worth $7·9 in 1950 and 8·6 lb. worth $7·4 in 1951. In 1951 24·3 lb. of full-strength toners were produced, a reduction of 4·7% on the 1950 figures. Over half the production, 14·6 lb., consisted of red toners. The five chief toners produced were Lithol Red (*C.I.* 189), 5·3 lb.; Toluidine Red (*C.I.* 69), 2·7 lb.; Benzidine Yellow and copper phthalocyanine, 1·6 lb. each; and Para Red (*C.I.* 44), 1·1 lb. Sales of full-strength toners were 24 lb. worth $30·1 in 1950 and 20·1 lb. worth $35 in 1951. Production of extended toners at 11·8 lb. was the same in 1951 as in 1950; sales in 1950 were 10·5 lb. worth $8·4 and in 1951 10·3 lb. worth $8·6.

C O C

Production of Carbon Black in the U.S.A. during 1951

Production in 1951 was (all weights in million lb.) 1677, i.e. 21% more than in 1950. Sales at 1563 increased by 4%. Stocks were 203 compared with 114. Production of furnace black was up by 35% and its sales by 16%. Sales of contact blacks were down by 12%. In 1951 furnace blacks were 62% of total sales compared with 56% in 1950. Exports increased by 8% during the year to a total of 433. In December 1951 there were 58 plants in production with a daily capacity of 5, compared with 4 at the same time in 1950.

C O C

Associação Brasileira de Química

The Associação Brasileira de Química has been formed by the merging together of the Sociedade Brasileira de Química founded in 1922 and the Associação Química do Brasil founded in 1941. It has a membership of about 1000.

C O C

Now only One Manufacturer of Oilcloth in the U.S.A.

The Coated Products Division of Interchemical Corporation ceased to make oilcloth on 30th June 1952, leaving only one manufacturer of this product in the U.S.A. The market has shrunk because of changes in the way of living, e.g. the trend toward hard-top tables, paper shelf edgings, etc.

C O C

New Books and Publications

The Chemistry of Synthetic Dyes
Volume I

By K. Venkataraman. Pp. xvi + 704. New York: Academic Press Inc. 1952. Price, 116s. 0d.

The publication of a treatise on the theory and technology of synthetic dyes has been long overdue, and is an event of profound importance to dye chemists in all branches of the industry.

The older text-books of Cain and Thorpe, Fierz-David, Georgievics, Wizinger, masterpieces in their time, are now classics. More than a generation has passed, and in this period new fibres and dyes of ever-improving properties have been developed. With laudable opportunism, fortified by the wealth of technical information from German sources available in *BIOS* and *FIAT* reports, Professor Venkataraman has compiled a comprehensive and critical survey of dye technology, combining the theoretical and practical aspects in even measure, and fulfilling the publishers' statement that— "the object has been . . . . to discuss the structure and synthesis of dyes against the general background of developments in the theory and practice of organic chemistry, ... and to stimulate academic and fundamental research . . . . ".

Critical selection from patent literature and technical communications is a herculean task, even for the industrial research worker in daily touch with recent developments, but the author has made a very realistic effort.

Volume I is now available and deals mainly with intermediates, azo dyes, and the effect of chemical constitution on colour. Volume II, dealing with vat dyes, the phthalocyanines, and triphenylmethane, azine, sulphur, and miscellaneous dyes, together with general topics such as light fastness, substantivity, and identification, is to be available later this year.

A quarter of the volume under review is rightly given over to the raw materials and organic intermediates of the dye and related industries. Coal tar constituents are dealt with at length, and a table is included containing 215 aromatic compounds present in coal tar arranged in order of boiling point. The technical aspects of the preparation, manufacture, properties, and toxic hazards are dealt with for an exhaustive list of intermediates. Although mainly for dye purposes, the range covers general organic chemicals of interest in perfumery, photography, and other non-dye uses. The various unit processes of sulphonation, halogenation, nitration, amination, and hydroxylation are reviewed adequately, and are followed by brief descriptions of the preparation and properties of individual intermediates.

There are altogether some 1800 references given in the book, of which about one-third appear in the chapter on intermediates.

The deliberate omission of an index at the end of Volume I is only partly mitigated by a full list of contents, so that searching for an individual intermediate or dye may sometimes be slow. For the sake of those interested in Volume I only, the inclusion of an index in a revised edition is imperative.

Some 50% of the volume deals with the vast group of azo dyes. Diazotisation methods and constitution and stabilisation of diazonium compounds are dealt with in sufficiency with recognition of the availability of specialist works on this subject.

The mechanism of coupling with a whole range of coupling component types and the constitution of azo dyes are treated theoretically in a very interesting and readable manner, as is the classification and the dyeing and fastness properties in a general chapter on azo dyes.

Separate chapters are devoted to monoazo and disazo dyes, mordant azo dyes, trisazo and polykisazo dyes, direct cotton dyes aftertreated on the fibre, dyes for cellulose acetate, pyrazolone dyes, and those derived from stilbene. The treatment of these chapters is distinguished by being precise in the constitution of the dye, adequate in the method of preparation and in the detail of fastness properties, and remarkable for the inclusion of so high a proportion of recent data. It may be criticised for the repeated tendencies to cataloguing, reminiscent of the style sometimes seen in *Annual Reports*, making for less interesting reading. The reviewer found very annoying the employment in these, and one or two other chapters, of roman numerals to avoid repetition of names in the text and in some of the tables. This practice may be perfectly justified in *Chemical Abstracts*, but is undoubtedly not in keeping with the character of a work of this magnitude.

The chapter on the constitution of metal-dye complexes forms a very interesting essay on this difficult subject, and follows the recent work given in the chapter on mordant azo dyes.

The whole of the technical aspects of azoic dyes and the preparation of Fast Bases, Fast Salts, and Naphtols are covered in the final chapter, which includes much valuable correlation of the effect of chemical constitution on fastness properties. This is one of the best chapters in the book, but it is marred in the final few pages by the off-hand manner in which are tabulated the constitutions, mainly from German information, of azoic pigments, occasionally in error and sometimes with repetition. Azo pigments are dealt with in one or two other parts of the book, but considering the scale of manufacture of this important group of synthetic dyes, and particularly their ever increasing employment for textile uses, such as in the mass-pigmentation of viscose and other fibres, and for pigment printing compositions, the author should have devoted at least one chapter to this subject.

Concise chapters are given to smaller groups of dyes— nitroso, nitro, thiazole, etc. The history and commercial aspects of dye manufacture in an early chapter are of interest. On pp. 402–403, in the chapter on nitroso dyes, the method of preparing Pigment Green B is not the best, nor is this

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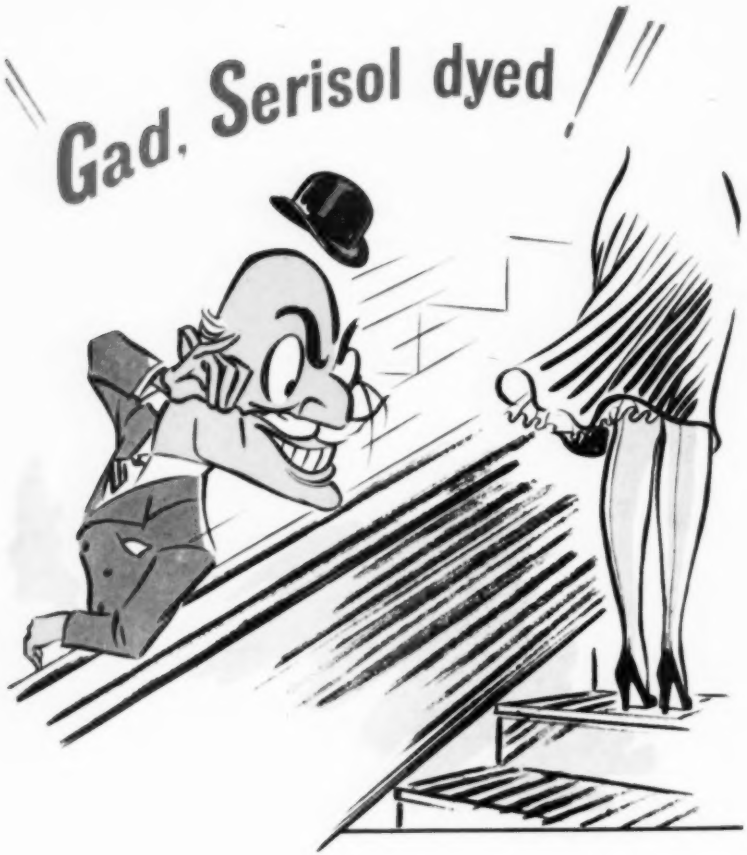

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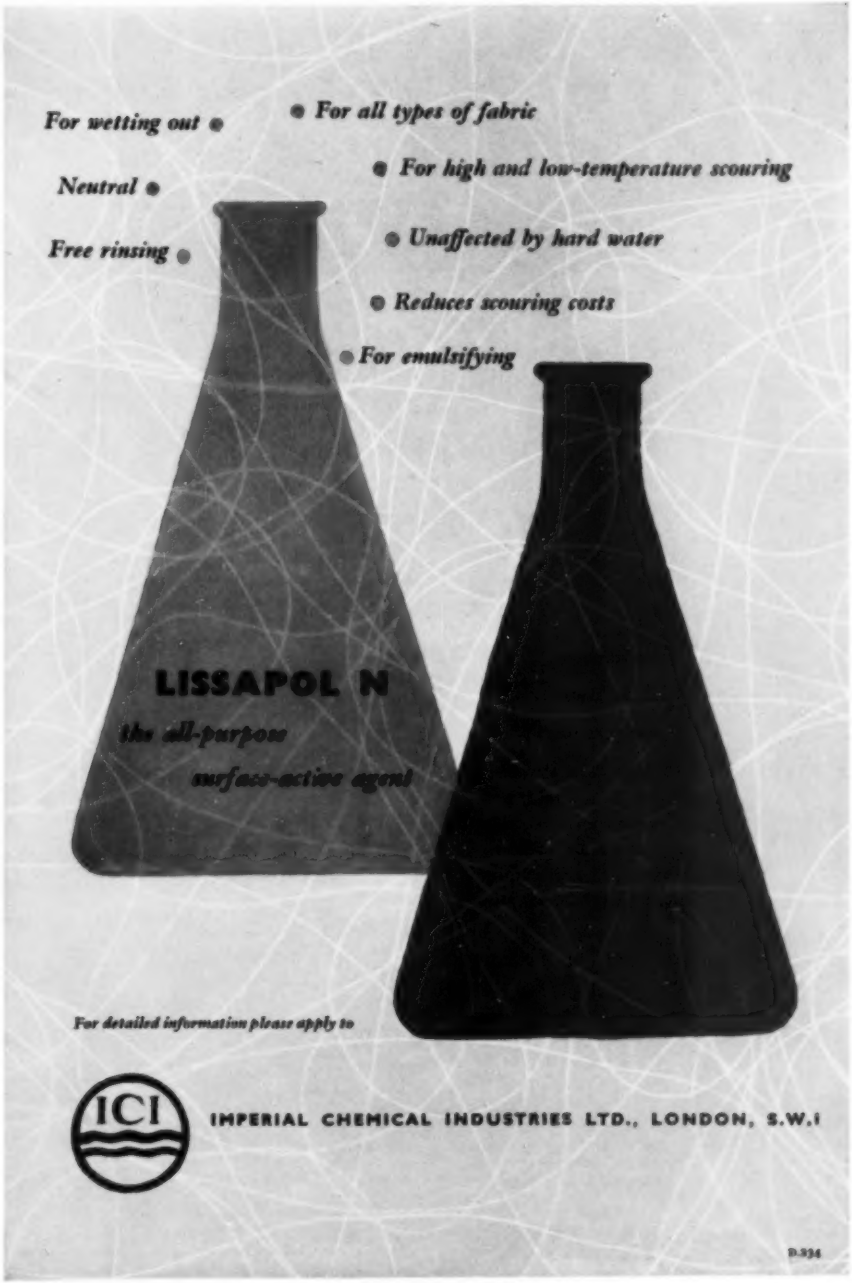
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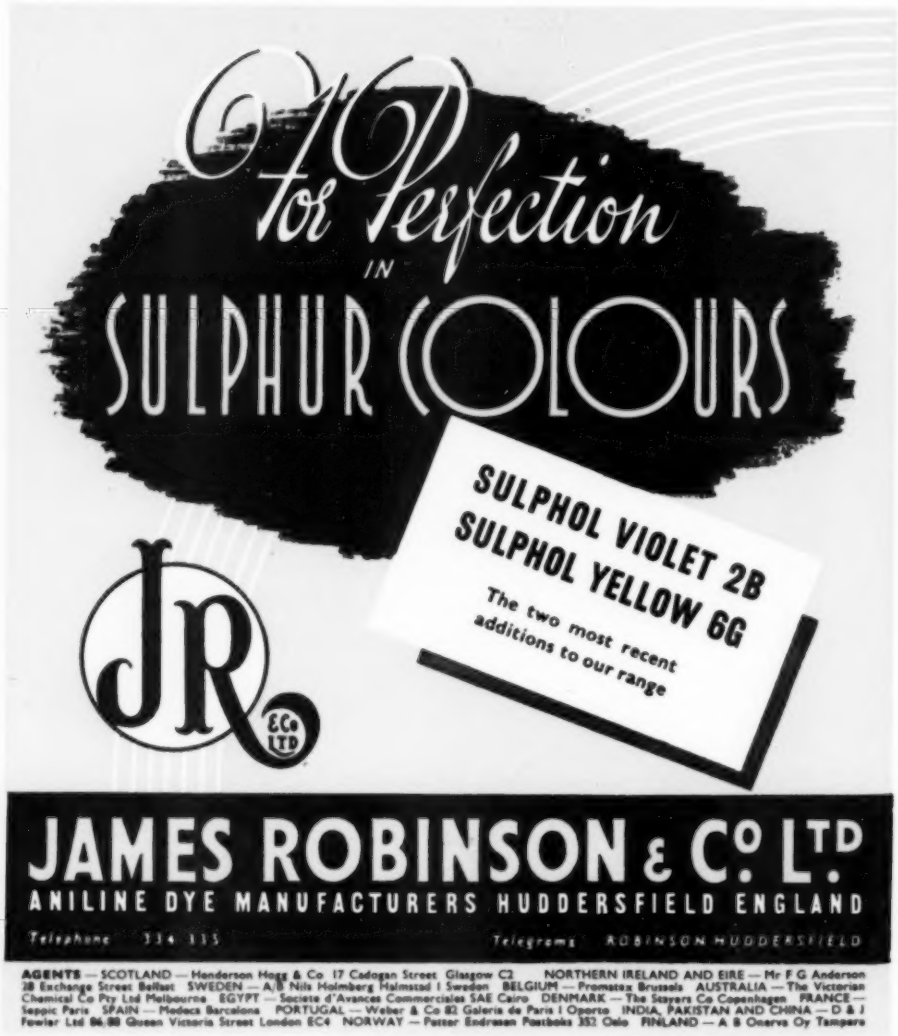

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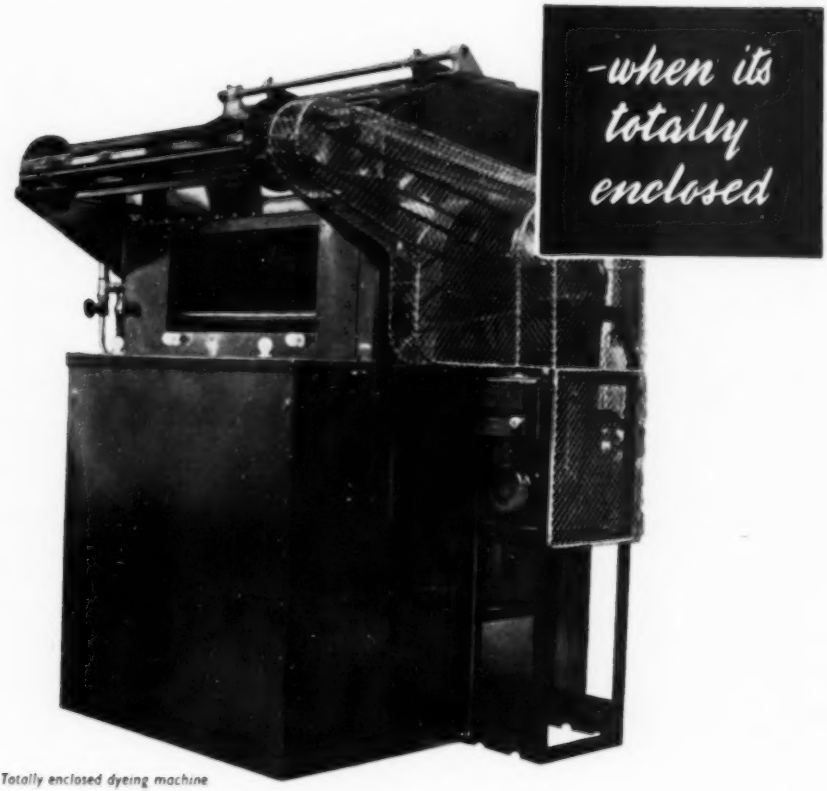

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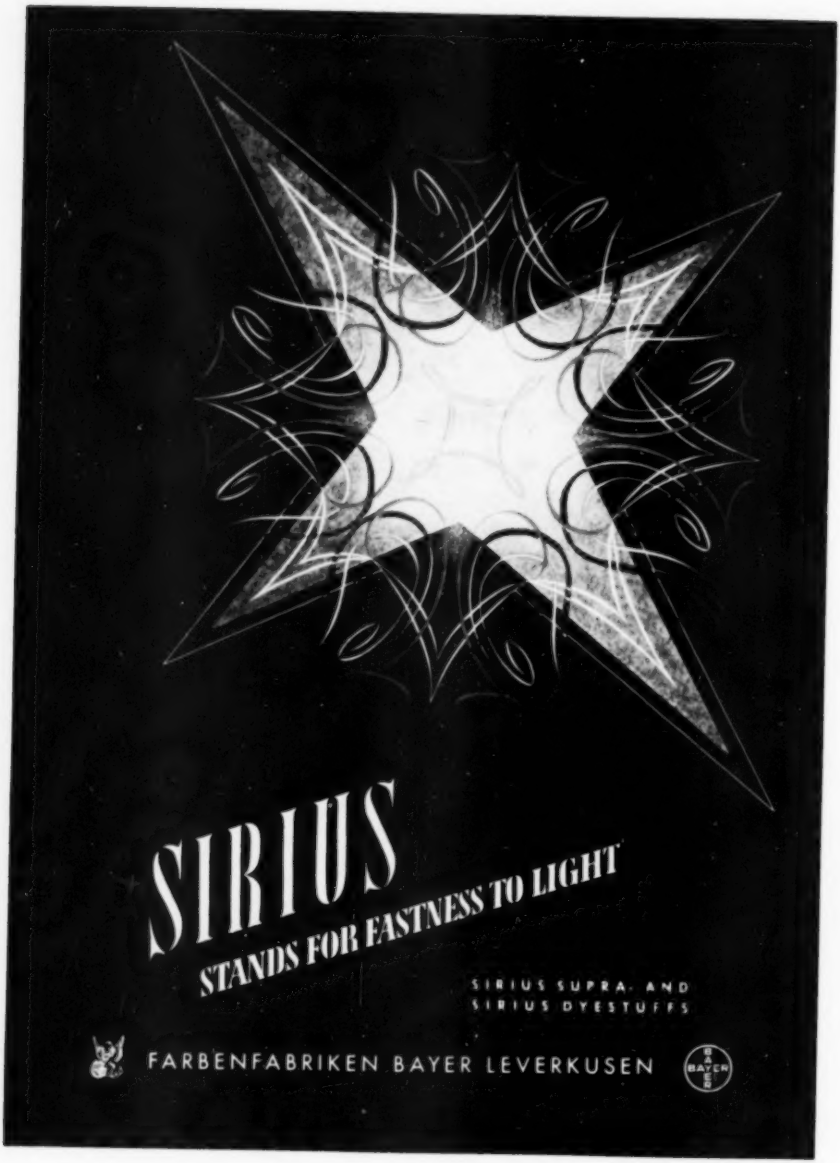

SIRIUS
STANDS FOR FASTNESS TO LIGHT
SIRIUS SUPRA- AND
SIRIUS DYESTUFFS
FARBENFABRIKEN BAYER LEVERKUSEN
BAYER

IRGALAN

a new name in wool dyestuffs

Bulk tested approved and adopted

combining

entirely new dyeing characteristics,

level results by short and simple dyeing process giving increased production rates,

enhanced spinning properties,

solid shades on "tippy" and blended wool,

outstanding general fastness,

compatibility.

Eight brands now available

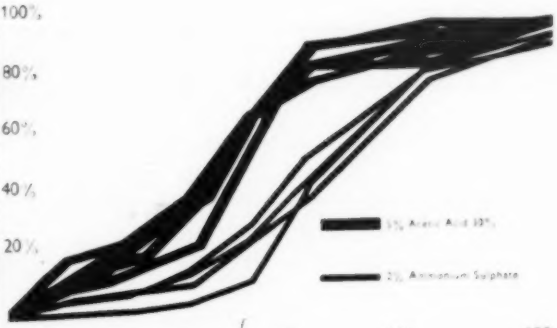
Geigy

MIGRATION

Loss of depth in dyed material

Increase in depth of undyed material

EXHAUSTION (Time and Temperature combined)



MINS	30	60	90	15	30	45	60	75	105	135
TEMP (C)		100°		40°	55°	70°	85°	100°	100°	100°

TYPICAL MIGRATION AND EXHAUSTION CURVES

Rhodes, Middleton, Manchester

THE GEIGY COMPANY LTD

Remember—

when dyeing wool

DURAMINE

AND

EVRON FAST

DYESTUFFS

Town End Chemical Works Limited

BRAMLEY • LEEDS

pigment used for "filling", but for pigmenting rubber. The reviewer can vouch for the correctness of the constitution of this pigment, which is normally given without the sodium atom. The numbering allocated to the bisulphite ester of nitroso-β-naphthol and to nitroso-Schaeffer acid has been confused.

Some sixty odd pages are given over to classification of dyes in groups and according to dyeing properties, and to lengthy descriptions of the various fibres and their identification.

Also interposed in the middle of the volume and preceded by a useful chapter on the measurement of colour from a physical standpoint there is a lengthy up-to-date critical review of published work on the effect of chemical constitution on colour. Extensively referenced and with ample extracts from work carried out during the last 10–15 years, this chapter on one of the most interesting topics of theoretical colour chemistry is without doubt one of the best yet to be seen in any book on dye chemistry, and will appeal to many who have found an interest in the, as yet unsolved, mystery of colour and its relation to chemical structure. The author acknowledges the criticism of Dr. L. G. S. Brooker on this topic. First, there is a brief treatment of the early theories of Witt (chromophores and auxochromes), Armstrong (quinonoid), Dilthey and Wizinger, and Stieglitz. This is followed by a discussion on the interpretation of absorption spectra, energy levels, and resonance theories, leading to Lewis and Calvin's theory based on the absorption of light and excitation of electron oscillations. The usual examples are given of the effect of geometry on light absorption, the effect of steric factors on resonance, the relationship between intensity of absorption bands and structure, and more recent work on the calculation of absorption spectra by quasi-classical and quantum-mechanical treatments. The azo chemist who notes that most of the examples are on hydrocarbons, polyenes, cyanines, and triphenylmethane dyes will perhaps be stimulated to consider his problems in the light of the theories so ably expounded for other compounds.

The book is well produced, as would be expected from the publishers, and for such a voluminous work amazingly free from typographical errors. Some points for consideration for revision have been given already. A further point is the method of suffixing references at the bottom of the page; for example, on pp. 530–531 there are six separate references to p. 523, reference to which only leads to a more remote reference. This system is too often repeated and is tiresome.

However, Professor Venkataraman can be assured that his treatise will have a place of merit wherever dye research and technology are practised, and he deserves the thanks and congratulations from those engaged therein for this invaluable contribution to the literature. J. GLASSMAN

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University.

Hexoran Co. Ltd.

FIBROFIX CONC. is a new, readily soluble brand of Fibrofix, of which only 2 lb. is used where 3 lb. of the old product was recommended. Besides being much stronger, Fibrofix Conc. has better fixation properties. The improved solubility is very advantageous, with regard both to the ease of preparation, and to the amount which can be handled conveniently in a single container.

Imperial Chemical Industries Ltd.

BASIC DYESTUFFS—This card shows twenty-four dyes of this type on cotton yarn. Where more than one brand of a particular dye is available, these are listed in groups against the relevant fastness data and dyed pattern. A number of basic dyes which are never applied to cotton in any of their forms are listed in the text together with their normal usage. In addition to information concerning their application to cellulosic fibres by both dyeing and printing techniques, details are given of methods of application to wool, silk, leather, bast fibres, paper, printing inks, and cellulose nitrate lacquers, for their use in colouring wood, carbon papers, and typewriter ribbons, and for producing toners and lakes by five different methods.

POLYMON PINK FFS—This is the first member of a range of dyes produced for the mass colouring of plastics and is primarily designed for pale pinks in polystyrene and urea–formaldehyde moulding powders, for which purpose pigment-type dyes must be used. It has good stability to heat and may be used in both opaque and highly translucent materials. It is also of interest for colouring cellulose nitrate and acrylic plastics but not those derived from phenol–formaldehyde or polyvinyl chloride. Some fastness grades include—*Polystyrene*—Light 2–3 (opaque and translucent), heat good. *Urea–formaldehyde*—Light 4–5, heat good.

SUPRA RED OXIDES LS, MS, DS—These are inorganic pigments giving in oil media red–browns which are richer and deeper than those obtained using Indian Red Oxides. They give shades having a fastness to light of 10+ on the 1–10 scale and their general high fastness properties make them particularly suitable for colouring cellulose nitrate lacquers, leathercloth coatings, and paints and for incorporation in aqueous leather finishes.

ULTRALAN LEATHER BROWN 6RS—This premetallised dye is of interest for producing a wide range of browns on all types of leather, particularly full chrome and mordant chrome gloving leather. Fastness figures include—Light 5, washing 3–4, solubility 3, penetration 1.

ULTRALAN ORANGE GRS—This premetallised dye has good levelling properties and is particularly useful as a component of mixtures for fast-to-wear shades on such materials as tropical suitings and gabardines. It is of interest for ground shades on wool for subsequent discharge printing, and suitable, but of only limited interest, for direct printing on wool and silk. Fastness figures include—Light 6–7, washing (repeated 5 times at 40°C.) 4, chlorination 4–5.

Abstracts from British and Foreign Journals and Patents

(The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952); while other abbreviations and symbols, together with a list of the periodicals abstracted, will be found at the end of the annual index.)*

I— PLANT; MACHINERY; BUILDINGS

Review of Dyes and Machinery suitable for Felts and Galloons. D. Haigh. *Dyer*, **107**, 709–712 (16 May 1952).

Report of a lecture in which a survey is made of fibres used in hat manufacture. Suitable machinery, dyes, and dyeing processes are discussed, and illustrations on the correction of processing faults given. Technical service provided by research associations is also summarised. A. H.

Use of Cathodic Polarisation for Preventing Corrosion in the Textile Industry. H. Weissbach. *Melliand Textilber.*, **33**, 445 (May 1952).

When bleaching with acid solutions of sodium chlorite in stainless steel baths, corrosion of the steel can be prevented by placing carbon electrodes at suitable points in the liquor, connecting these to the positive pole of a 3·5 v. D.C. supply, and connecting the walls of the bath to the negative pole. About 1 amp. per sq. m. of bath surface is required, and this is attained with a carbon electrode surface of ~ 200 sq. cm./amp. In this method, the electrodes are not attacked, and hence continual replacements are not required and there is no contamination of the soln. It is therefore preferable to methods in which no external source of current is used, pieces of aluminium or magnesium being immersed in the bath and connected externally to the walls. A. E. S.

Ultrasonics in the Textile Industry. J. Salquain. *Teintex*, **17**, 377–389 (June 1952).

The production of ultrasonic radiations is described together with their application in the textile industry, including the dyeing of hydrophobic fibres such as nylon and Orlon, production of stable emulsions, preparation of aqueous sols of waxes and other water-insol. products, preparation of aqueous dispersions of oil-sol. dyes, homogenisation of glues and finishes, homogenisation of dye pastes and dispersions, increase in rate of scouring and dyeing, oxidation of dyes, production of non-shrink wool, and the continuous dyeing of wool. B. K.

Continuous Fibre Dyeing. S. H. Williams. *Amer. Dyestuff Rep.*, **41**, P 360–P 364 (9 June 1952).

Description of a machine (patented) for a continuous dyeing process for raw stock, sliver, staple fibre, and the like. It is based on the well known Williams unit modified to allow the passage through the unit of two mesh belts, between which the fibre mass is held. Several units in tandem and various types of multiple dyeing operations are described. Spray pipes between the dyeing units permit washing or application of chemical solutions; five ton squeezing nips are also inserted between each unit. A. H.

PATENTS

Thread-storage, Thread-advancing Reels. Courtaulds. *BP* 674,604

A reel is described in which formation of barre effects is avoided on thread being treated at high speeds. C. O. C.

Insert for Freshly Spun Rayon Cakes. Algemene Kunstzijde Unie. *BP* 674,535

The inside of a freshly spun viscose rayon cake is protected during washing, etc. by inserting a rectangle of permeable cloth, e.g. preshrunk cotton, with stainless steel wire sewn on the top and bottom edges. W. G. C.

Dielectric Heating of Rayon Cakes. Westinghouse Electric Corpn. *USP* 2,586,328

Vibrations as an Aid in Scouring. J. E. Excell and H. K. Jones. *BP* 673,250

Apparatus whereby the washing liquor is vibrated comprises two hollow bodies flexibly connected to each other in superposed coaxial relation. An electrically operated device is associated with one of the hollow bodies to impart vibration of 50–200 cycles/sec. to both. The vibration is constrained to axial vibration in opposite directions. The hollow bodies are placed in the washing liquor. Better cleansing is obtained without any tendency to cause wool to felt. C. O. C.

Attachment to Dyeing Machines for Piling Warps in a Box or Tub. T. Cook and T. A. Crabtree. *BP* 674,006

A reciprocating winch attached to the machine feeds the warp lengthwise in the box. Associated reciprocating peg guides distribute the warp about the breadth of the box in even layers. The mechanism can be adjusted to various sizes of warps. C. O. C.

Drying Warps. Bachmann Uxbridge Worsted Corpn. *BP* 674,329

Imparting Dimensional Stability to Tubular Knit Fabric. F. Chatfield and F. R. Redman. *USP* 2,583,630

The fabric is fed at a predetermined speed to a given point, following which the speed of the fabric is decreased and simultaneously the fabric is stretched sideways to cause its stitches to expand transversely to the wales, after which it is moistened and pressed to set the stitches. C. O. C.

Squeegee Mechanism for Screen Printing Machines. Meyercord Co. *BP* 675,181

The squeegee is lifted off the screen during the return stroke. A scoop picks up ink at the supply end of the screen at the finish of the printing stroke, and deposits it ahead of the squeegee at the other end of the screen, opposite the supply of ink for use during the printing stroke and for return to the ink supply by the squeegee. *BP* 675,257

A manually operated clutch enables the squeegee to be stopped at the end of its printing stroke. C. O. C.

Ager. R. W. Jacoby. *BP* 673,582

An ager is described in which local heating is prevented by directing a large volume of steam on to the fabric where the reaction commences, so that the heat given out is absorbed by the steam without its temperature rising unduly. C. O. C.

Felting Hoods for Hats. M. Cause. *BP* 674,479

A felting machine for hoods has at least three beds of rollers co-operating in pairs. A conveying mechanism receives the articles as they emerge from the first pair of rollers and delivers them to the second pair. This mechanism changes the position of the articles in relation to the beds of the rollers as it carries them from one pair of beds to the next. C. O. C.

Ingredient-dispersing Apparatus for Laundry Washing Machines. Prosperity Co. *BP* 674,376

Spotting Fabrics with Organic Solvents and Recovery of the Solvent. American Steel Foundries. *USP* 2,586,569

Apparatus is described whereby fabric can be spotted by forcing dry cleaning fluid through it with automatic reclamation of the cleaning fluid. C. O. C.

Nylon Hosiery Mending Apparatus. M.K.M. Hosiery Mills. *BP* 674,209

The hose is extended over a mending cup, and following the repairing operation may be pressed between a pad and a heated iron whilst still in this position. G. E. K.

Drying Wallpaper. J. Dungler. *BP* 675,388

The paper is drawn through the drying chamber by externally arranged rewinding means and is extended in the chamber as a flat surface in several independent elements. High-speed jets of hot air and/or superheated steam are directed perpendicularly against these flat surfaces so as to press the paper against them. This permits quick drying without cockling. C. O. C.

Treating Regenerated Cellulose Films. La Cellophane. *BP* 674,266

Provision is made for ensuring constant tension even when the film shrinks in length during processing. C. O. C.

Continuous Milling of Plastics. British Resin Products. *BP* 675,112

Infrared Radiation Dryers. Baker Perkins. *BP* 673,956

Uniform treatment throughout the surface of the article is obtained by causing air currents to flow downwards in opposition to the convection currents, thus preventing overheating of that portion of the goods in the upper part of the dryer. C. O. C.

Producing Coloured Design in Hosiery or other Non-woven Textile Fabrics (IX p. 369).

II—WATER AND EFFLUENTS

Application of Chlorine in the Control of Microbiological Organisms in Process Waters. S. H. Newland. *Amer. Dyestuff Rep.*, **41**, P 392–P 397 (23 June 1952).

Chlorination of water for textile purposes performs two functions— control of sliming organisms and destruction of organic matter. The chlorine remaining may be present as chloramines, dependent on the N_2 content of the water, or as hypochlorous acid, which is governed by pH. pH < 6·5 produces HOCl, pH 6·5–7·5 approx. equal quantities of HOCl and OCl^-, and pH 9·5–10 OCl^- alone. The active slime-killing agent is HOCl, hence the importance of pH control. Various types of organisms are discussed, including algæ, fungi, and bacteria. Iron and sulphur bacteria are especially to be avoided in textile waters. An amperometric residual chlorine recorder is described. A. H.

Organic Complexing Agents in Textile Processing. J. J. Singer and F. C. Bersworth. *Amer. Dyestuff Rep.*, **41**, P 365–P 370 (9 June 1952).

A brief description of the reaction between metallic ions and various organic complexing agents, followed by a discussion of the sequestering action of Versenes (Na ethylenediaminetetra-acetates). Wool scouring, prevention of iron stains in kier boiling and mercerising, stabilisation of H_2O_2 bleaching solutions, non-precipitation of Fe and hard-water salts in hypochlorite bleaches, and inactivation of metal impurities in dyebaths are discussed in detail. A. H.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Chain Mechanism of Decomposition of Hydrogen Peroxide and Existence of the Radical HO_2 and of A. N. Bakh's Higher Peroxide of Hydrogen. K. E. Kruglyakova and N. M. Emanuel. *Doklady Akad. Nauk S.S.S.R.*, **83**, 593–596 (1 April 1952).

In 1897 A. N. Bakh postulated the formation of the HO_2 radical during the decomp. of hydrogen peroxide and suggested also that two such radicals may combine forming the higher peroxide H_2O_4. A summary is given of some work that supports these hypotheses. The kinetics of the thermal decomp. of hydrogen peroxide solutions is examined, the reaction being followed by measuring the vol. of oxygen evolved and by titrating cooled samples for residual H_2O_2. The reaction rate constantly increases during the early stages, and a branched-chain mechanism is postulated. In general, the vol. of oxygen evolved is less than that calculated from the titre of the residual H_2O_2, but complete decomp. is found to give a vol. of oxygen somewhat greater than that calculated. It is considered that HO_2 radicals accumulate during the decomposition in appreciable amount and that, on cooling a sample for titration, the dimer H_2O_4 is formed. H_2O_4 titrates as H_2O_2, but yields a greater amount of oxygen when it decomposes. Some H_2O_4 is present already in the original hydrogen peroxide and accounts for its high oxygen yield. Other possible explanations of the S-shaped reaction curve, e.g. supersaturation of the soln. with oxygen, autocatalysis, leaching out of catalysts from the silica vessel during the reaction, etc., are considered and rejected for various reasons. In agreement with the hypothesis, freshly distilled hydrogen peroxide solutions decompose more slowly on heating than the same soln. does after being preserved cold for a month (when slight decomp. leads to the formation of some H_2O_4). Addition to a hydrogen peroxide soln. of preparations believed to be rich in H_2O_4, e.g. the product of the action of atomic hydrogen on molecular oxygen, has a similar stabilising effect. A. E. S.

Preparation of Orthotitanic Esters by Transesterification. B. A. Arbuzov and Z. G. Isaeva. *J. Gen. Chem. U.S.S.R.*, **22**, 566–567 (April 1952).

Patent claims that the higher esters of orthotitanic acid may be prepared by the action of the alcohol on the acid in presence of dimethylaniline are not substantiated, but the n-butyl, n-hexyl, n-octyl, and n-nonyl esters are obtained in good yields by warming tetraethyl orthotitanate with the appropriate alcohol and distilling off the ethanol as it is liberated. A. E. S.

Tannin of the Pods of Tara cultivated in Italy. G. A. Bravo. *Cuoio, Pelli, Mat. concianti*, **28**, 25–32 (1952); *Chem. Abs.*, **46**, 4831 (25 May 1952).

Pods of tara from Sardinia contain 40–50% tannin; results of using tara tannin as a mordant for basic dyes in dyeing and printing are described. C. O. C.

PATENTS

Detergent. Marchon Products. *BP* 674,896

A mixture of a sulphated or sulphonated detergent compound neutralised with an organic base, a hydrophilic colloid, and a fatty-acid hydroxyalkylamide is stable to heat and is unaffected by hard water. C. O. C.

Detergent. Swift & Co. *USP* 2,586,496

In preparing detergents by condensing hydroxyalkylamines having at least one acylatable H atom in the amino group with carboxylic acids an improved product is obtained when the reaction product contains both the alkanolamide and the alkanolamine monoacid ester. This is brought about by using only primary and secondary alkanolamines. The best results are obtained when the acylating substance contains 8–14 C. C. O. C.

Detergent. Victor Chemical Works. *USP* 2,586,897

The product obtained by treating a dodecyl acid phosphate with an olefin oxide and then mixing with an alkali-metal salt of a molecularly dehydrated phosphoric acid in the proportion 1–75 organic product : 99–25 inorganic phosphate is a very efficient detergent. C. O. C.

Cationic Auxiliary Agents. Société Anonyme d'Innovations Chimiques dite: Sinnova ou Sadic. *BP* 674,953

Compounds of formula Alk·O·Ar(R)·CH_2·B·H]Cl⁻ (R = Alk of > 7 C; Ar may be subst. or unsubst.; B = a tertiary aliphatic amine which may contain one or more OH groups, or a tertiary phenolic amine, or a tertiary heterocyclic base) are less sensitive to Ca and Mg ions than sulphated fatty alcohols and possess similar detergent, wetting, etc. powers. C. O. C.

Increasing the Fastness to Washing of Direct Dyes. Ciba. *BP* 673,711

Dyeings or prints made with direct dyes containing sulpho or carboxyl groups are given improved fastness to washing by treating them with an aqueous solution of a condensate of an aldehyde with a compound containing at least one $-N{:}C(N<)_2$ group prepared in presence of 0·1–0·9 equiv. of acid per mol. of N compound, e.g. 1 mol. of dicyandiamide condensed with 2–3 mol. of formaldehyde in presence of 0·16–0·5 mol. of acetic acid. C. O. C.

Organosilicon Compounds—Water-repellent and Foam-preventing Agents. Dow Corning. *BP* 673,139

Compounds of formula R·[SiR_2·CH_2]$_x$·SiY_2·R (R = Alk or monocyclic Ar; Y = Alk·O or Hal; x = 1 or 2) are of use as water-repellent or foam-preventing agents. C. O. C.

Long-chain-substituted Phenol-Aldehyde Resin Emulsion for Water-repellent Coatings on Nylon and Polyvinyl Fabrics. U.S. Secretary of War. *USP* 2,582,230

Resins formed from components one of which contains a long-chain Alk of > 11 C when applied to textiles impart very good water-repellency, affect neither handle nor strength, and are resistant to washing and dry cleaning. C. O. C.

Proteinaceous Adhesive, Sizing, and Coating Compositions. American Cyanamid Co. *BP* 674,129–31

A composition which readily disperses in water to yield stable, smoothly flowing sizes, etc. consists of an alkali-

solubilised protein and 5–30% (on wt. of dry protein) of a compound of formula R·NH·C(NH)·NH·CN (R = H, Alk of 1–4 C, or monohydroxyalkyl of 2–4 C) or of an ammonium or alkali-metal salt of dicyanimide or dicyanoguanidine. C. O. C.

Coating Compositions. Monsanto. *BP* 673,849

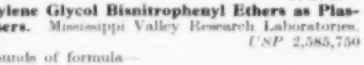
Hard, flexible, solvent-resistant films are given by compositions containing as the film-forming component > 50% of a copolymer of a secondary alkyl (4–12 C) half-ester of an ethylene-$\alpha\beta$-dicarboxylic acid with a compound of formula R·CH:CH_2 (R = subst. or unsubst. phenyl) and < 50% of a urea–aldehyde, alkylated urea–aldehyde, or amino aldehyde thermosetting condensate. C. O. C.

Insect Repellents. Sinclair Refining Co. *USP* 2,587,957

Compounds of formula $R^1{\cdot}CO{\cdot}NR^2R^3$ (R^1 = Alk containing at least one Hal; R^2 and R^3 = H or Alk), e.g. a chlorobutyramide, are white and crystalline. They are used to render textiles, paper, etc. insect-repellent. C. O. C.

Polyethylene Glycol Bisnitrophenyl Ethers as Plasticisers. Mississippi Valley Research Laboratories. *USP* 2,585,750

Compounds of formula—



(X = Br or Cl; m = 4–29; n = 0–3) are plasticisers for organic materials. C. O. C.

Polymerised Methyl Vinyl Ketone Latices. Celanese Corpn. of America. *BP* 674,653

The aqueous dispersion obtained by polymerising methyl vinyl ketone in a non-solvent aqueous medium in absence of dispersing agents, the initial weight ratio of water to ketone being at least 3 : 1, has many uses, including those of an adhesive and a coating agent, the sizing of paper, and for imparting wash-fast stiff finishes to textiles. C. O. C.

Vulcanisation Agents. B.B. Chemical Co. *BP* 674,391

Treatment of a zinc dithiocarbamate with a thiuram sulphide yields a vulcanisation agent which works at room temperature in absence of an accelerator or free sulphur. Such agents are readily soluble in organic solvents, and vulcanise rubber without detectable evolution of H_2S. C. O. C.

Synthetic Tanning Agents. Stalinovy Zavody. *BP* 675,478

Formalin (not > 10% H·CHO) is heated with a polyhydric phenol (all of whose phenolic OH groups are attached to the same aromatic nucleus) and with alkaline metal sulphites, any SO_2 formed is neutralised, and the mixture is evaporated under reduced pressure at not > 135° C. until a water-soluble product remains. C. O. C.

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Sulphonation Reaction. XXII—Preparation of Naphthalenetrisulphonyl Chlorides. A. A. Spryskov. *J. Gen. Chem. U.S.S.R.*, **22**, 661–667 (April 1952).

Following work on the action of chlorosulphonic acid on 1:5-naphthalenedisulphonyl chloride (see *J.S.D.C.*, **68**, 219 (June 1952)), the behaviour of a further five disulphonyl chlorides (1:3, 1:6, 1:7, 2:6, 2:7) is examined. The 1:3- and 1:7-dichlorides yield mixtures of trichlorides, but, on pouring the reaction mixture on to ice, the 1:6-, 2:7-, and 2:6-dichlorides yield trichlorides (1:3:6 from 1:6 and 2:7, and 1:3:7 from 2:6) that do not need further purification. Yields are increased by adding ~ 0·1 mol. of pyrosulphuryl chloride per mol. of chlorosulphonic acid. In this method of sulphonation, isomerisation of di- and trisulphonyl chlorides or sulphonic acids during reaction does not appear to occur. A. E. S.

3-Nitro-o-cresol. R. Long and K. Schofield. *J.C.S.*, 2437–2438 (June 1952).

A modification of Deninger's and Astle and Cropper's method is described for the isolation of 3-nitro-*o*-cresol as follows—o-Toluidine is diazotised in dil. H_2SO_4 by addition of ~ 50% aq. $NaNO_2$, and the solution poured as rapidly as gas evolution permits into H_2SO_4 (50% vol./vol.), initially at 140° C., the temperature during the decomposition being kept above 70° C. throughout. The black tarry liquid is steam-distilled, the 3-nitro-*o*-cresol collected, dried, distilled under reduced pressure, and the fraction of b.p. 99–104° C. at 7–8 mm. collected. 3:5-Dinitro-*o*-cresol is also isolated from the distillation. The formation of nitric oxide is observed during the reaction. H. H. H.

3-Chloro-4:6-dinitrobenzoic Acid. H. Goldstein and R. Stamm. *Helv. Chim. Acta*, **35**, 1330–1333 (June 1952).

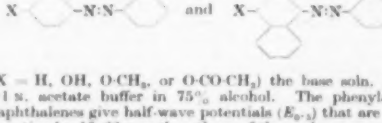
When *m*-chlorobenzoic acid is dissolved in conc. H_2SO_4, heated to 40° C., treated with potassium nitrate, the temperature rising to 65–70° C., and further treated with this reagent at 105–110° C., with final heating at 140–145° C., 3-chloro-4:6-dinitrobenzoic acid is obtained. Only mononitration has previously been reported by direct nitration of *m*-chlorobenzoic acid. H. H. H.

4:5- and 3:6-Dinitronaphthalic Anhydrides. F. Bell. *J.C.S.*, 1952–1953 (May 1952).

Various anomalies in the descriptions extant of the nitration of naphthalic anhydride are claimed to be resolved, and the preparations of 4:5- and 3:6-dinitronaphthalic anhydrides described anew. It is shown that these anhydrides can be heated with conc. HNO_3 for a considerable time without change. H. H. H.

Polarographic Investigation of certain Hydroxyazo Compounds. I. F. Vladimirtsev and I. Ya. Postovsky. *Doklady Akad. Nauk S.S.S.R.*, **83**, 855–858 (21 April 1952).

In polarographic determinations with a dropping mercury cathode and a calomel reference electrode in 0·0001 M. solutions of—



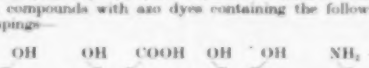
(X = H, OH, O·CH_3, or O·CO·CH_3) the base soln. is a 0·1 M. acetate buffer in 75% alcohol. The phenylazonaphthalenes give half-wave potentials ($E_{\frac{1}{2}}$) that are less negative by 13–22 mv. than those of the azobenzenes; i.e. they are more readily reduced. The effects of the substituents are similar in the two series—substitution of H by OH leads to a much less negative $E_{\frac{1}{2}}$ (by ~ 60 mv.), substitution by O·CH_3 has a smaller effect (~ 40 mv.), and substitution by O·CO·CH_3 has an inappreciable effect. The results show that in phenylazo-α-naphthol the quinone hydrazone tautomer is present in negligible amount, for the latter, if present, would be detected on account of its much less negative $E_{\frac{1}{2}}$ ($E_{0\cdot5}$ for the *N*-methyl deriv. of the quinone hydrazone is − 217 mv., while for phenylazo-α-naphthol it is − 365 mv.). Further experiments have been made with *para*- and *meta*-substituted phenylazonaphthols (α and β). In both α- and β-naphthol derivatives, replacement of *p*-H by O·CH_3 makes $E_{\frac{1}{2}}$ more negative by ~ 40 mv., and replacement of *p*-H by SO_3H makes $E_{\frac{1}{2}}$ less negative by 20 mv. Replacement of *m*-H makes $E_{\frac{1}{2}}$ less negative by 10 mv. in each case. All these changes can be readily interpreted in terms of the electron-donating and -accepting characters of the various groups, and the polarographic method is thus shown to be capable, in certain cases, of characterising in an approximately quantitative fashion the mutual interactions of groups within molecules. A. E. S.

Chemistry of 4-Nitro-4′-aminodiphenyl Sulphone. III—Products of Coupling the Diazo Compound with Nitrogen-containing Derivatives of Phenols and Naphthols and with Sulphonic Acids of Phenols, Naphthols, Naphthylamines, and Aminonaphthols. M. S. Zhedek, S. S. Shtal, and M. A. Gorinshteyn. **IV—Products of Coupling the Diazo Compound with Sulphanilamides.** M. S. Zhedek and M. A. Gorinshteyn. *J. Appl. Chem. U.S.S.R.*, **25**, 441–449, 449–451 (April 1952).

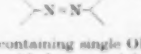
4-Nitro-4′-aminodiphenyl sulphone is diazotised and coupled with a great variety of substances to give 65 azo dyes (62 of them new). The properties of the dyes, including their antituberculer potency and their colour in acid and alkaline soln. and as dyeings on wool and silk, are tabulated. A. E. S.

Nickel Derivatives of Azo Dyes. N. M. Mackenzie, H. E. Millson, and B. L. West. *Ind. Eng. Chem.*, **44**, 1017–1023 (May 1952).

Nickel, like Cr, Co, Fe, and Cu, is able to form co-ordination compounds with azo dyes containing the following groupings—



and also with dyes containing single OH and NH_2 groups *ortho* to azo, but not with dyes containing the grouping—



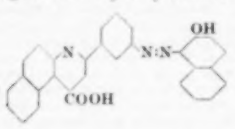
The complex may be applied to the fibre in the form of a premetallised dye, or formed on the fibre by aftertreatment with Ni salt. On wool the Ni derivatives differ in colour from the corresponding Cr derivatives, and generally possess inferior wet fastness, but many have outstanding fastness to light. Ni-containing azo substantive dyes and pigments also have excellent fastness to light. W. K. R.

Scattering of Light in Colloidal Dye Solutions. S. R. Sivarajan. *Current Sci.* (India), **20**, 202–203 (1951): *Chem. Abs.*, **46**, 4886 (10 June 1952).

Light scattering in aq. Chrysophenine G (*C.I.* 365) (0·066 g./litre) containing 8 g. NaCl per litre at 26–85°c. corresponds to that expected for anisotropic molecules that are small with respect to the wavelength of the incident white light, and increases in both size and isotropy with increasing temperature. Aq. Purpurine 4B (*C.I.* 448) (0·082 g./litre) containing 12 g. NaCl per litre at 27°c. contains anisotropic micelles that are large with respect to the wavelength of the light scattered. As the temperature rises to 50°c. the micelles apparently grow in only one direction, but above 50°c. (the transition temperature for this dye and salt concentration) the micelles tend to go into solution and the scattering decreases. C. O. C.

Doebner Reaction. XVII— 2-*m*-Nitrophenyl-5:6-benzoquinoline-4-carboxylic Acid. N. Gallo. *Gazz. Chim. ital.*, **81**, 327–331 (1951): *Chem. Abs.*, **46**, 5047 (10 June 1952).

The preparation of the above acid from β-naphthylamine, *m*-nitrobenzaldehyde, and pyruvic acid is described. Reduced with $FeSO_4$ and coupled with β-naphthol, it yields an orange–red wool dye of probable formula—



C. O. C.

Dyes having Antipyrin Nuclei— II. O. F. Ginzburg and B. A. Poray-Koshits. *J. Gen. Chem. U.S.S.R.*, **22**, 715–718 (April 1952).

The dye obtained by oxidising diantipyrylphenylmethane (see *J.S.D.C.*, **66**, 68 (Jan. 1950)) is converted into its carbinol, which, unlike the carbinols of triphenylmethane dyes, is appreciably sol. in water and may be titrated in soln. A slightly alkaline soln. of the carbinol is titrated potentiometrically with acid. Conversion of the carbinol into the dye with liberation of hydroxyl ions takes an appreciable time, and 3–4 min. is required after each addition of acid for an equilibrium potential to be attained. One equiv. of acid is required for the neutralisation of 1 mole of carbinol, and the apparent basic dissociation const. of the carbinol is found to be $10^{-7\cdot4}$. A. E. S.

Metachromasia in Dyes. P. Schlechter and M. Campani. *Arch. Sci. biol.* (Italy), **32**, 165–182 (1948): *Chem. Abs.*, **46**, 5111 (10 June 1952).

Solutions of dyes rendered metachromatic by adding chromotropic substances, e.g. hyaluronic acid, lose the metachromasia and resume their original colour on dilution or addition of acids, salts, or organic solvents. Adsorption on $Al(OH)_3$ separates the dye from the chromotropic substance. In saline solution the metachromatic dyes are positively charged; after adding the chromotropic substance the charge becomes negative, and changes again to positive on restoring the original colour by the above method. For a dye to be metachromatic it must have an imino-*p*-quinone nucleus and be capable of changing to a mesomeric form with an unsaturated atom bound to the nucleus. A large elongated molecule with weak oxyacid and secondary alcoholic functions is characteristic of chromotropic substances (acid polysaccharides). C. O. C.

Internuclear Cyclisation. V— Cyclisation of Derivatives of Diphenylmethane, Benzophenone, and *N*-Methyldiphenylamine. D. H. Hey and R. D. Mulley. *J.C.S.*, 2276–2287 (June 1952).

Diazonium compounds prepared from 2-aminodiphenylmethane, 2-aminobenzophenone, 2-amino-2′-nitrobenzophenone, 2-amino-*N*-methyldiphenylamine, and 2-amino-*N*-methyl-2′-nitrodiphenylamine are converted by a variety of methods (e.g. action of heat or of copper powder on the aqueous solutions) into the fluorene, fluorenone, and *N*-methylcarbazole systems respectively. Examples have been established in which the formation of the new internuclear bond involves the ejection from a nitrophenyl group of (*a*) nuclear hydrogen at the *meta*-position to the nitro group, and (*b*) the nitro group itself. H. H. H.

Xanthones and Thioxanthones. IV— Preparation and Properties of 9-Oxa-1-aza-anthrone and 9-Thia-1-aza-anthrone. F. G. Mann and J. A. Reid. *J.C.S.*, 2057–2062 (June 1952).

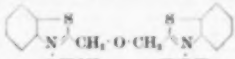
An improved preparation of 2-chloronicotinic acid is described starting from quinolinic anhydride, which is converted by gaseous ammonia into 2-carbamylnicotinic acid, thence by the Hofmann reaction to 2-aminonicotinic acid, followed by an improved diazotisation process to the hydroxy acid, and finally to the chloro compound by the method of Fibel and Spoerri; this cyclised with $POCl_3$ to give 9-oxa-1-aza-anthrone, and, when heated with free thiophenol, it gave 3-carboxy-2-pyridyl phenyl sulphide, which was cyclised with $POCl_3$ to give 9-thia-1-aza-anthrone. The basic properties of the thia compound, although weak, are stronger than those of the oxa analogue. Both anthrones are reduced to the corresponding anthranols when treated with mercury and sodium in ethanolic solution. H. H. H.

Some Benziminazole Derivatives. B. N. Feitelson, P. Mamalis, R. J. Moualim, V. Petrow, O. Stephenson, and B. Sturgeon. *J.C.S.*, 2389–2398 (June 1952).

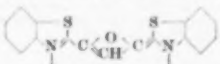
117 Polymethylenebisbenziminazoles, phenyl- substituted benziminazoles, and alkyl- and chloroalkylbenziminazoles have been synthesised and their properties are now recorded. H. H. H.

αγ-Epoxythiacarbocyanines— I. I. K. Ushenko. *J. Gen. Chem. U.S.S.R.*, **22**, 711–715 (April 1952).

Condensation of diglycollyl dichloride with *N*-ethyl-*o*-aminothiophenol led to the formation of the compound—



This was converted into the iodide, which was then treated with orthoformic ester to yield the new dye—



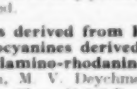
The corresponding diphenyl cpd. was prepared in a similar manner. The absorption maxima of these dyes differ very little from those of the corresponding known dyes containing $-CH_2-$ in place of $-O-$, but their stability to light and to alkalis is much less. Attempts using an analogous method of preparation to produce dyes containing $-S-$ or $-S\cdot S-$ in place of $-O-$ were unsuccessful— sulphur was eliminated, and the products were known trimethin dyes. A. E. S.

***mero*Cyanines derived from Thio-oxindole. I—*mero*Cyanines having a Dimethin Chain attached to the 3-Thionaphthen Nucleus.** R. H. Glauert and F. G. Mann. *J.C.S.*, 2135–2144 (June 1952).

By condensing thio-oxindole-3-aldehyde with the quaternary salts of various heterocyclic compounds having a suitable reactive methyl group, a new type of *mero*-cyanine has been prepared in which the thionaphthen nucleus is linked at the 3-position to the dimethin chain. Since certain of these compounds are active photographic sensitisers, a wide range has been prepared by variation of (*a*) the type of heterocyclic compound, (*b*) the alkyl groups used for the quaternisation, and (*c*) the nature and position of substituents in either or both of the heterocyclic systems present in each *mero*cyanine. Further variation has been obtained by the insertion of methyl groups in the α- and the β position in the dimethin chain. For comparative purposes, one compound has been prepared in which the two heterocyclic systems are directly joined, with the dimethin chain thus eliminated. H. H. H.

***mero*Cyanine Dyes derived from Rhodanine. III—Dimethin*mero*cyanines derived from 3-Amino- and 3-Diacetylamino-rhodanine.** Z. P. Sytnik, S. V. Natanson, M. V. Deychmeyster, and L. D. Zhilina. *J. Gen. Chem. U.S.S.R.*, **22**, 705–711 (April 1952).

Eleven members of a new group of dimethin*mero*-cyanines derived from 3-diacetylaminorhodanine are synthesised, the constant residue being—



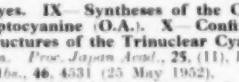
and the other residue being e.g. a thiazole analogue or quinoline. The new dyes have higher solubility and better sensitising properties than the corresponding aminorhodanine or unsubstituted rhodanine cyanines, but there is little difference between the absorption maxima of corresponding members of these three groups of dyes. A. E. S.

Oxonols derived from Azol-5-ones. E. B. Knott. *J.C.S.*, 2399–2401 (June 1952).

Oxonols (dyes obtained by linking two heterocyclic keto-methylene nuclei by a methin group) are in many instances poor photographic sensitisers, which may be due partly to their poor adsorption on the grain but more probably because steric hindrance prevents coplanarity of the two nuclei. A number of symmetrical and unsymmetrical oxonol dyes derived from 2-substituted thiazol-5-ones and 2-phenyloxazol-5-one are now described. The reaction of 4-ethoxymethylene-2-phenyloxazol-5-one and 2-benzyl-thiothiazol-5-one leads to a colourless chromotropic product which is isomeric with the required oxonol, and which sensitises a silver halide emulsion to visible light in the same way as related normal oxonols. H. H. H.

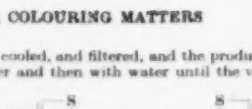
Cyanine Dyes. IX—Syntheses of the Components of Cryptocyanine (O.A.). X—Confirmation of the Structures of the Trinuclear Cyanine Dyes. T. Ogata. *Proc. Japan Acad.*, **28**, (11), 17–21 (1950): *Chem. Abs.*, **46**, 4531 (25 May 1952).

IX—Lepidine ethiodide (I) with triethyl orthoformate (II) in presence of acetic anhydride yielded cryptocyanine (O.A.) 2 (III), which reacted again with (II) to produce cryptocyanine (O.A.) 1 (IV) and with I to produce crypto-cyanine (O.A.) 3 (V). (V) was produced in 22% yield by boiling 0·24 g. (III), 0·3 g. (I), and 1 ml. acetic anhydride for 1 hr.; cryptocyanine (O.A.) 4 (VI) was obtained by extracting the filtrate with water.



(V)

X—Tetramethyltrimethinthiazolocyanine iodide (VII) and 0·1 ml. (II) in 0·5 ml. acetic anhydride kept at b.p. for 20 min. gave 6% thiazolocyanine (O.A.) 1 (VIII). (VII) (1 g.), 0·1 g. 2-(2 anilinovinyl)-5-methylthiazole methiodide, and 0·1 g. K acetate in 0·5 ml. acetic anhydride were boiled for 1 hr., cooled, and filtered, and the product was washed with ether and then with water until the violet colour of

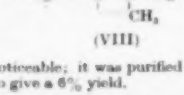


(VIII)

(VIII) was noticeable; it was purified by recrystallisation from water to give a 6% yield. C. O. C.

Effect of Intramolecular Hydrogen Bonds on the Colour of Indoaniline Dyes derived from α-Naphthol. B. S. Portnaya, I. I. Levkoev, and N. S. Spasokukotsky. *Doklady Akad. Nauk S.S.S.R.*, **82**, 603–605 (1 Feb. 1952).

Thirteen dyes of formula—



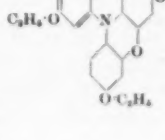
are prepared, and the absorption spectra of their alcoholic solutions are determined. When R is an electronegative radical, there is the expected bathochromic shift, attributable to an electron shift towards the quinone oxygen, from the spectrum of the parent cpd. (R = H). When R is incapable of hydrogen bonding with the quinone oxygen (R = Cl, $COOCH_3$, $CO{\cdot}N(C_2H_5)_2$, $CO{\cdot}N(CH_3){\cdot}C_6H_5$, $CO{\cdot}N(C_6H_5)_2$), the effect is generally small—11–17 mμ., apart from $COOCH_3$ (35 mμ.)—but when such hydrogen bonding can occur (R = COOH, $CO{\cdot}NH_2$, $CO{\cdot}NH{\cdot}C_2H_5$, $CO{\cdot}NH{\cdot}C_6H_5$), the shift is much greater (112, 55, 53, 75 mμ. respectively). For R = $SO_2{\cdot}NH{\cdot}C_2H_5$, $SO_2{\cdot}N(C_2H_5){\cdot}C_2H_5$, and $SO_2{\cdot}N(C_2H_5)_2$, the shifts are approx. equal (55, 55, and 63 mμ. respectively), and it is considered that steric effects prevent the first of these groups from participating in hydrogen-bond formation. A. E. S.

Mechanism of Wohl's Reaction for the Preparation of Phenazine. E. I. Abramova and I. Ya. Postovsky. *J. Gen. Chem. U.S.S.R.*, **22**, 502–509 (March 1952).

The reaction of nitrobenzene with aniline in presence of caustic alkali yielding phenazine, phenazine oxide, and *p*-nitrosodiphenylamine was considered by Wohl to result from the isomerisation of nitrobenzene into *o*- and *p*-nitrosophenol. The formation of azobenzene as a by-product is not explained by Wohl's scheme. It is now shown that neither unstable *o*-nitrosophenol nor the stable analogue 1-nitroso-2-naphthol will react with aniline under the conditions of Wohl's reaction. It is considered that hydroxyl ions, or other suitable anionic reagent, attack nitrobenzene by addition at the nitro group, giving a cpd. that reacts with aniline to give azobenzene, or by "unorthodox" *ortho-para* substitution in the nucleus, leading to phenazine (and its oxide) and *p*-nitroso-diphenylamine. Sodamide has been found to be a suitable condensing reagent for the Wohl reaction, and in this case the formation of an addition cpd. at the nitro group has been demonstrated. Sodium anilide is now shown to be another suitable condensing agent. A. E. S.

***p*-Phenetidine Brown.** T. Pavolini, F. Gambarin, and W. Manzini. *Ann. Chim.* (Rome), **40**, 647–653 (1950): *Chem. Abs.*, **46**, 4550 (25 May 1952).

Evidence is given suggesting that the structure of *p*-phenetidine brown (obtained from *p*-phenetidine and 3% H_2O_2 in dil. H_2SO_4) is—

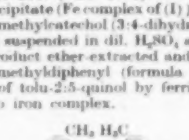


C. O. C.

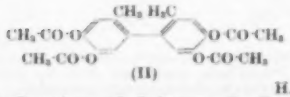
Absorption Spectra and Dissociation Constants of Diphenylthiocarbazone (Dithizone) and its Internal Complex Salts—I and II. R. E. Chernutskaya. *J. Gen. Chem. U.S.S.R.*, **22**, 403–408, 408–414 (March 1952).

Oxidation of 4-Methylcatechol in Aqueous Solution by Ferric Ion. H. Burton and H. B. Hopkins. *J.C.S.*, 2445–2447 (June 1952).

The black precipitate (Fe complex of (I)) from the mixing of aq. soln. of 4-methylcatechol (3:4-dihydroxytoluene) and ferric acetate in suspended in dil. H_2SO_4 and treated with SO_2, and the product ether-extracted and acetylated to a tetra-acetoxydimethyldiphenyl (formula (II) suggested). The oxidation of tolu-2:5-quinol by ferric ions gave the quinone and no iron complex.



(I)



(II)

H. H. H.

Addition Reactions of Quinones. I—Reaction of Cysteine and Thiourea and its Derivatives with some Quinones. H. Burton and S. B. David. *J.C.S.*, 2193–2196 (June 1952).

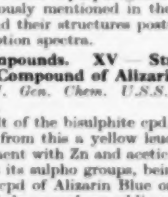
The formation of additive compounds from various quinones and cysteine, thiourea, and *N*-arylthioureas is investigated. 2-Methyl-1:4-naphthaquinone (2 mol.) and cysteine (1 mol.) are found to give the highly coloured *S*-3-methyl-1:4-naphthaquinon-2-ylcysteine, in a slightly acid medium. In aq. HCl, benzoquinone, tolu-2:5-quinone, and 1:2-, 1:4-, and 2-methyl-1:4-naphthaquinone all give genuine 1:1-additive compound hydrochlorides with thiourea, although the salt from 1:4-naphthaquinone is hydrated. Under suitable conditions *S*-2:5-dihydroxyphenylthiourea undergoes hydrolysis to a hydroxybenzothioxolone, which can be hydrolysed further to mercaptoquinol. H. H. H.

Methyl Ethers and Acetates of Purpurogallin. G. D. Thorn and L. R. C. Barclay. *Canadian J. Chem.*, **30**, 251–256 (April 1952).

A number of acetates of purpurogallin and of its methyl ethers, not previously mentioned in the literature, have been prepared and their structures postulated from their ultraviolet absorption spectra. C. H. R.

Bisulphite Compounds. XV—Structure of the Bisulphite Compound of Alizarin Blue. V. N. Ufimtsev. *J. Gen. Chem. U.S.S.R.*, **22**, 658–661 (April 1952).

The pure K salt of the bisulphite cpd. of Alizarin Blue is prepared, and from this a yellow leuco cpd. is formed in soln. by treatment with Zn and acetic acid. The leuco cpd. readily loses its sulpho groups, being converted into the brown leuco cpd of Alizarin Blue on boiling, or into the red salt of this leuco cpd. on adding alkali in the cold. The absorption spectra are determined of solutions of the bisulphite cpd. and of its leuco form. On the basis of the experimental results and of the known behaviour of hydroxyquinolines, the widely held hypothesis that bisulphite reacts with the quinone carbonyls is rejected, the proposed formula for the bisulphite cpd. being—



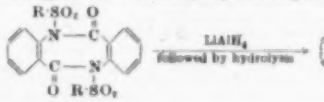
A. E. S.

Sulphenic and Selenenic Acids and their Derivatives. II—1-Methoxyanthraquinone-4-selenenic Acid. W. Jenny. *Helv. Chim. Acta*, **35**, 1429–1434 (June 1952).

In contrast to the results of Fries and Schürmann on the halogenation of 1:1'-dimethoxydianthraquinonyl 4:4'-disulphide, the bromination of 1:1'-dimethoxydianthraquinonyl 4:4'-diselenide proceeds normally, with the formation of 1-methoxyanthraquinonyl 4-selenobromide. The preparations of ethyl 1-methoxyanthraquinone-4-selenenate, the corresponding selenenic acid, and 1-methoxyanthraquinonyl-4-selenyl acetate are described. H. H. H.

Action of Lithium Aluminium Hydride on (*a*) *NN'*-Diarylsulphonyl Derivatives of Dianthranilide, and (*b*) Photo-peroxides of 9:10-Diarylanthracene. A. Mustafa. *J.C.S.*, 2435–2437 (June 1952).

When benzene solutions of *NN'*-diarylsulphonyl derivatives (I (*a*)–(*c*)) of dianthranilide are treated with excess of lithium aluminium hydride, the corresponding *o*-arylsulphonamidobenzyl alcohols (II (*a*)–(*c*)) are obtained in almost quantitative yield. The action of lithium aluminium hydride on the photo-peroxides (III (*a*)–(*c*)) of 9:10-diarylanthracenes, in ether–benzene, yields (IV (*a*)–(*c*)) respectively in almost quantitative yield. The action of lithium aluminium hydride on (I) is an example of the cleavage of the C–N linkage by this reagent.



(I) (II)

(*a*) R = C_6H_5; (*b*) R = *p*-$C_6H_4CH_3$; (*c*) R = *o*-$C_{10}H_7$

(III) (IV)

(*a*) R = C_6H_5; (*b*) R = *m*-$C_6H_4CH_3$; (*c*) R = *p*-$C_6H_4CH_3$

H. H. H.

Synthesis of Cyananthrene. W. Bradley and F. K. Suteliffe. *J.C.S.*, 2118–2122 (June 1952).

The *iso*-structure of cyananthrene is confirmed by a synthesis from 3-bromopyridino (3′:2′-8:9) *meso*benzanthrone under conditions which convert 3-bromo*meso*benzanthrone into isoviolanthrone. Its structure, confirmed by its absorption spectrum in conc. H_2SO_4, is therefore dipyridino(2′:3′-3:4)(2″:3″-12:13)*iso*violanthrone—

H. H. H.

Thio-oxindole, Thioindoxyl, and certain Carbonyl Derivatives. R. H. Glauert and F. G. Mann. *J.C.S.*, 2127–2135 (June 1952).

By boiling a solution of the 3-hydrazone of thionaphthenquinone in 25% aq. KOH under reflux for several hours, and acidifying to precipitate *o*-mercaptophenylacetic acid, the latter on steam distillation from HCl soln. gives thio-oxindole in 90% yield. Three methods are described for the preparation of thio-oxindole-3-aldehyde, while 3-acetyl(thio-oxindole) and 2-acetyl(thioindoxyl) are readily prepared by the action of *NN'*-diphenylacetamidine on thio-oxindole and thioindoxyl respectively. H. H. H.

Structure of Indigo and its Derivatives. H. von Eller. *Acta Cryst.*, **5**, 142 (1952): *Chem. Abs.*, **46**, 4315 (25 May 1952).

The lattice constants and space groups (all = $P2_1/a$) are given for indigo, thioindigo (two modifications), and selenium indigo. Patterson projections of Se indigo (modification I) yield the Se parameters. The heavy-atom method gives a Fourier projection that shows the molecule to be the *trans*-ethylenic isomer. C. O. C.

Biogenesis of Flower Pigments. L. Reichel. *Forschungen und Fortschr.*, **26**, Sonderheft 2, 5–6 (1950): *Chem. Abs.*, **46**, 4610 (25 May 1952).

In a brief survey the importance of the C_{15} series in the formation of flower pigments is indicated as well as the

fundamental rôle of the 1:3-diphenylpropane structure. Of the possible mechanisms—(I) $C_6{-}C_3 \rightarrow C_6 \rightarrow C_6{-}C_2{-}C_6$, (IIa) $C_6{-}C_3 + C{-}C_3 \rightarrow C_3{-}C_2{-}C_6$, and (IIb) $C_6{-}C + C_3{-}C_6 \rightarrow C_6{-}C_2{-}C_6$, (IIa) and (IIb) have been established. Components are mainly hydroxyacetophenones, *ar*-hydroxy-*o*-hydroxyacetophenones, and hydroxybenzaldehydes, with aldol condensations followed by expulsion of H_2O and reduction. With the exception of the chalkones, ring formation determines the various compounds. The catalytic rôle of amino acids is stressed. C. O. C.

Constitution of Hypericin. H. Brockmann, E. H. von Falkenhausen, and A. Dorlars. *Naturwissenschaften*, **37**, 540 (1950): *Chem. Abs.*, **46**, 5034 (10 June 1952).

It has previously been shown that hypericin is either a hexahydroxy-2:2′-dimethylnaphthodianthrone or a hexahydroxy-2:2′-dimethylhelianthrone (*Chem. Abs.*, **37**, 5053 (1941)). Reductive acetylation of hypericin gave a blue compound, the hepta-acetyl derivative of 2:2′-dimethylnaphthodianthrene. It was confirmed that hypericin has four hydroxy groups in α-positions (*Chem. Abs.*, **45**, 2462, 3369 (1951)). The hepta-acetyl compound has absorption bands of shorter wavelength than hypericin, likewise acetoxynaphthodianthrene compared with 2:2′-dihydroxynaphthodianthrone. This shift of bands, unusual for acetyl compounds, is explained by steric hindrance of the acetyl groups attached to the 2 and 2′ C causing increased excitation energy of the ring system. It seems that in hypericin this same steric hindrance exists after acetylation, and hence that the other two hydroxyl groups are in the 7- and 7′-positions (*cf.* numbering in *Chem. Abs.*, **45**, 2462 (1951)). C. O. C.

Fuerstiaquinone. P. Karrer and C. H. Eugster. *Helv. Chim. Acta*, **35**, 1139–1147 (June 1952).

The behaviour towards acids, bases, and various reagents of fuerstiaquinone, the red pigment of formula $C_{20}H_{14}O_8$ extracted by Fries from *Fuerstia africana*, is described. In particular, it forms a dark green copper complex with copper acetate which is soluble in chloroform and benzene, thereby indicating the presence of an *o*-hydroxyquinonoid grouping. H. H. H.

Thermal Cyclisation of *o*-Aroyloxyacetoarones. II—Further Syntheses in the Flavone Series (Diflavones and Flavonols). H. M. Lynch, T. M. O'Toole, and T. S. Wheeler. *J.C.S.*, 2063–2067 (June 1952).

The method for the preparation of flavones involving thermal cyclisation in glycerol of aroyl esters of *o*-hydroxyacetoarones is now applied to the production of diflavones and flavonol 3-methyl ethers, by using, respectively, esters of 4:6-diacetylresorcinol and of *o*-methoxyphloroacetophenone. H. H. H.

Rearrangement in the Demethylation of 2′-Methoxyflavones. E. M. Philbin and T. S. Wheeler. *Chem. and Ind.*, 449 (17 May 1952).

2′:5′-Dimethoxyflavone, treated with HI in phenol at 170–180° C. under pressure, rearranges completely to 6:2′-dihydroxyflavone; at 160° C. and atmospheric pressure the rearrangement is partial. J. W. B.

Chlorophyll. A. Stoll and E. Wiedemann. *Fortschr. Chem. Forschungen*, **2**, 538–608 (1952): *Chem. Abs.*, **46**, 5142 (10 June 1952).

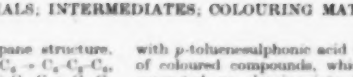
Review. 435 references. C. O. C.

Corymbiferin, a Pigment derived from a Glucoside occurring in *Gentiana corymbifera*. D. J. Ross. *New Zealand J. Sci. Technol.*, **32 B**, (3), 39–43 (1950): *Chem. Abs.*, **46**, 4539 (25 May 1952).

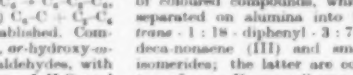
Data are given of the properties of this pigment, which occurs in the roots of this plant; it has the composition $C_{15}H_{10}O_7$ and a trihydroxydimethoxyxanthone structure. C. O. C.

Carotenoid Syntheses. IX—Stereoisomeric 1:18-Diphenyl-3:7:12:16-tetramethyloctadeca-nonaenes. C. H. Eugster, C. Garbers, and P. Karrer. *Helv. Chim. Acta*, **35**, 1179–1189 (June 1952).

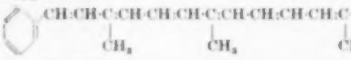
1-Phenyl-3-methylhexa-1-en-5-yn-3-ol (I) is prepared from benzylideneacetone, propargyl bromide, and magnesium, and when heated with anhydrous oxalic acid *in vacuo* gives 6-phenyl-4-methyl-hexa-3:5-dien-2-one (II). Condensation of (I) with octa-4-en-2:7-dione gives a tetrol, which on partial reduction and subsequent heating with *p*-toluenesulphonic acid in toluene affords a mixture of coloured compounds, which are chromatographically separated on alumina into the main reaction product *trans*-1:18-diphenyl-3:7:12:16-tetramethyloctadeca-nonaene (III) and small amounts of three *cis*-isomerides; the latter are converted by iodine into the *trans*-form. Very small amounts of another pigment were isolated.



(I)



(II)



H. H. H.

Use of Nitro- and Halogeno-ketones in the Synthesis of Pteridines, including Pteroic Acid, from 2:4:5-Triamino-6-hydroxypyrimidine. F. E. King and P. C. Spensley. *J.C.S.*, 2144–2152 (June 1952).

Derivatives of Naphthaquinone. IX—Pigments from Sea Urchins 4. C. Kuroda and M. Okajima. *Proc. Japan Acad.*, **26**, (6), 33–36 (1950): *Chem. Abs.*, **46**, 4518 (25 May 1952).

A bright red pigment, 2:3:6:8-tetrahydroxy-1:4-naphthaquinone, has been extracted from the spines of *Anthocidaris crassispina*. Spinochrome B, found in the spines of other sea urchins, is 2:3-dihydroxy-6:8-dimethoxy-1:4-naphthaquinone. C. O. C.

Pigment Colors for Plasticised Vinyl Chloride Polymers. G. Wormald and W. F. Spengeman. *Ind. Eng. Chem.*, **44**, 1104–1107 (May 1952).

Polyvinyl chloride (P.V.C.) is invariably coloured by pigments because of the low fastness of oil-soluble dyes. Synthetic pigments are classified, and the properties and suitability for colouring P.V.C. of each group are outlined. Inorganic pigments are generally more stable to light and heat, particularly in pale shades, and have better resistance to bleeding and migration than organic pigments. Consideration must be given, however, to the effect of the metal constituent of the pigment on the polymer, plasticisers, *etc.* Thus Fe and Zn can promote rapid degradation by heat and light, and Mn or other oxidation catalyst may promote rancidity in the final product. Acid-sensitive sulphide pigments require careful stabilisation to avoid odours. Of the organic pigments, phthalocyanine blue and green give excellent results. Azo dye lakes, e.g. Lithol Rubine, are generally superior in resistance to bleeding and migration, and often in resistance to light and heat, to the insoluble azo dyes, e.g. Toluidine Red, but their poor alkali resistance necessitates careful selection of the polymer stabiliser. The insoluble azo dyes are mostly unsuitable for colouring P.V.C. Lakes derived from acid and basic dyes are also unsuitable owing to poor light fastness and bleeding characteristics. Many vat dye pigments are satisfactory, but some have poor resistance to migration and to rubbing. W. K. R.

Mechanism of Drying Retardation by Toluidine Red Toners. A. Underhang. *Paint*, **22**, 254–255 (July 1952).

The retardation of drying by Toluidine Red toners has two causes—firstly presence of free β-naphthol and secondly the method by which the toner was ground. To reduce this defect to a minimum and so enable red enamels to be made that will dry as quickly as those of other colours, the Toluidine Red toner should be washed free from β-naphthol, and very low temperatures should be maintained during drying and grinding of the toner. C. O. C.

Composition of Prussian Blue by Thermometric Method. R. S. Saxena and A. K. Bhattacharya. *J. Indian Chem. Soc.*, **28**, 703–709 (Dec. 1951).

In continuation of previous papers on the composition

of Prussian Blue by physicochemical methods, thermometric titrations between ferric chloride and potassium ferrocyanide at several concentrations have been made. In the direct titration ($FeCl_3$ to $K_4Fe(CN)_6$) curves, two breaks are observed, corresponding approximately to the formation of $KFe[Fe(CN)_6]$ and $Fe_4[Fe(CN)_6]_3$, whereas in the reverse titration ($K_4Fe(CN)_6$ to $FeCl_3$) there is one break only, corresponding to formation of $KFe[Fe(CN)_6]$. Observed titre values in the direct method are slightly higher than theoretical, and in the indirect titration slightly lower. This is explained by the tendency of the compound formed to hydrolyse, and addition of alcohol, by suppressing hydrolysis, produces titre values closer to the theoretical. The results obtained are in conformity with results from conductimetric and potentiometric experiments. A. H.

Oil Black. W. H. Shearon, R. A. Reinke, and T. A. Ruble. *Ind. Eng. Chem.*, **44**, 685–694 (April 1952).

A detailed description is given of modern technique in the production of carbon black by the combustion of mineral oil in refractory furnaces. A thermally cracked oil of high aromatic content is sprayed into the furnace, and a mixture of air (50% of that required for complete combustion) and natural gas (to provide heat for cracking) is introduced tangentially. A higher yield is obtained than in other methods of carbon black manufacture. The product is of fine particle size and imparts high abrasion resistance to rubber. It is used chiefly in tyre manufacture. W. K. R.

PATENTS

1:4-Naphthaquinones. Eastman Kodak Co. *USP* 2,585,229

4a:5:8:8a-Tetrahydro-1:4-naphthaquinones* and 5:8-dihydro-1:4-naphthaquinones are readily and cheaply oxidised to 1:4-naphthaquinones by an oxidising agent, e.g. nitrous or nitric acid, in presence of a mineral acid. C. O. C.

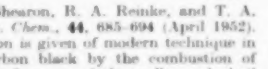
* Carbon atoms 4a and 8a are common to the two benzene nuclei.

1-Aminoaryl-3-methyl-5-pyrazolones. Ciba. *BP* 673,623

1-Aryl-3-methyl-5-pyrazolones containing NH_2 in the aryl nucleus are prepared by condensing an aminoarylhydrazine with acetoacetamide. Thus an aq. soln. of acetoacetamide, made by adding diketen to ice-cold aq. NH_3, is stirred for 2–3 hr. at room temp. with an aq. soln. of *m*-aminophenylhydrazine dihydrochloride containing sodium acetate. The resulting pyrazolone is precipitated by adding NaCl. R. K. F.

Colour Couplers. Gevaert. *BP* 674,103

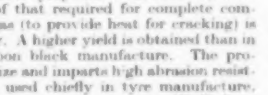
Compounds of formula—



(D = atoms to complete a 5- or 6-membered ring; X = radical obtained by eliminating one H of a hydroxylamine, hydrazine, hydrazone, or hydrazide) yield dyes with primary aromatic amino developers. C. O. C.

Colour Couplers. Ilford. *BP* 674,356

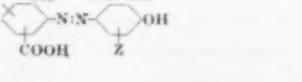
Compounds of formula—



(R^1 = an aromatic residue which may contain one or more sulphonic acid radicals; R^2 = Alk of 10–18 C; R^3 = phenyl or naphthyl containing a *p*-OH group) are colour developers for use with aromatic primary amino developers. C. O. C.

Metal-complex o-Amino-o'-hydroxyazo Compounds —Dyes for Nylon. General Aniline. *USP* 2,583,925

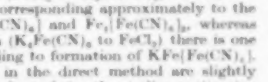
Metal complexes of monoazo compounds—



(the COOH is *meta* or *para* to the azo link; Y = H, Alk, O·Alk, Hal, or NO_2; Z = H, Alk, O·Alk, or Hal; R = H or Alk) are sufficiently water-soluble to be applicable to nylon without use of a dispersing agent. Thus, all parts being by weight, 3-aminosalicylic acid hydrochloride (19) is diazotised and coupled with *NN*-diethyl-*m*-aminophenol in presence of NaOH and Na_2CO_3. Excess alkali is neutralised with acetic acid, conc. aq. ammonia (130–175) added, and then $CoCl_2.6H_2O$ (24). After stirring for 4 hr. at 50–60°C. the cobalt complex is filtered off and converted to its Na salt with aq. NaOH. It dyes nylon orange-red from an acid bath. E. S.

Brown, Metal-complex (Copper), Stilbene Azo Direct Cotton Dyes. Ciba. *BP* 674,708

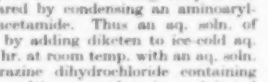
By condensing 4:4'-dinitrostilbene-2:2'-disulphonic acid with one or two primary amino compounds, at least one of which is an aminodisazo dye containing an *oo'*-dihydroxyazo, *o*-carboxy-*o'*-hydroxyazo, or *o*-hydroxy-*o'*-methoxyazo grouping, copperable stilbene azo compounds—



(Z = azo or azoxy; R^1, R^2, and R^3 = aromatic residues of < 11 aryl C atoms, R^3 containing no OH; of R^1 and R^3 one contains OH *ortho* to the azo link, and the other COOH, O·CH_3, or OH *ortho* to the azo link; R^4 = an organic radical bound by an aryl residue to Z) are produced. The copper complexes formed from them are brown direct cotton dyes. Thus, all parts being by weight, the aminodisazo dye (52) 2-aminophenol-4:6-disulphonic acid →(1) Acid →(2) *m*-toluidine is boiled with disodium 4:4-dinitrostilbene-2:2'-disulphonate (24) in water (1200) containing NaOH (30). After addition of HCl and NaCl, the dye is filtered off and treated at 90–95°C. with copper sulphate (50) dissolved in water (1100) containing ammonia (40 of 25%) and monoethanolamine (5), to produce the copper complex, which dyes cotton brown-violet. E. S.

Blue, Metal-complex (Copper), Direct Cotton Dyes. Ciba. *BP* 674,767

Blue, substantive dyes are made by coppering in substance disazo dyes—



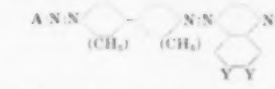
(R = an *ortho*-coupling naphthol, which may contain 1 or 2 SO_3H groups) under such conditions that splitting up of the O·Alk groups takes place. A particularly valuable dye is obtained by coupling tetrazotised dianisidine, first with 1 mol. of 1-naphthol-3:8-disulphonic acid, and then with 1 mol. of 2-*p*-carboxymethoxyphenylamino-5-naphthol-7-sulphonic acid in presence of Na_2CO_3, and finally coppering. The coppering may be carried out with copper acetate at > 100°C., e.g. by drying and heating at about 125°C. the reaction mixture or the copper complex primarily produced (i.e. the compound of copper and dye in which no splitting up of O·CH_3 groups has yet taken place). E. S.

Disazo Direct Cotton Dyes. General Aniline. *USP* 2,584,935

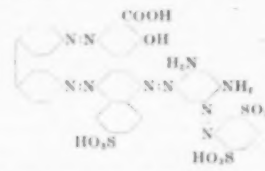
Yellow, orange, and brown direct cotton dyes, suitable for aftertreatment on the fibre with copper salts, are made by coupling tetrazotised 4:4'-diamino-3:3'-dicarboxydiphenyl-urea or -thiourea with one mol. of an acetoacetanilide containing a sulpho group in the anilide nucleus and with one mol. of an acetoacetanilide containing no water-solubilising substituent in the anilide nucleus. Thus, tetrazotised 4:4'-diamino-3:3'-dicarboxydiphenyl-urea is coupled with a solution containing 1 mol. of acetoacetanilide and 1 mol. of *o*-chloro-*p*-sulphoacetoacetanilide dissolved in aq. NaOH, in presence of Na_2CO_3. The dye is isolated as a sodium salt, and then converted to the free acid by treatment with acetic acid before drying. Finally it is mixed with soda ash, disodium phosphate, and Nekal BX. The resulting composition has good solubility in water, and dyes cotton yellow, improved in wash fastness by treatment on the fibre with aq. copper sulphate; it does not stain acetate rayon. The coppered dyeings are dischargeable to pure white by alkaline hydrosulphite. E. S.

Brown, Tetrakisazo Dyes for Leather. Gy. *BP* 674,291

Tetrakisazo dyes which dye leather dark brown with little penetration are made when the diazo compound of an aminodisazo dye—



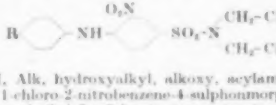
(A = an *o*-hydroxybenzoic acid; one Y = H, the other Y = SO_3H), and the diazo compound of an aniline-disulphonic acid, are coupled with *m*-phenylene(or tolylene)diamine. They have good solubility in a formic acid dyebath, and hence may be applied together with a penetrating acid dye in presence of formic acid. Thus, tetrazotised benzidine is coupled, first under alkaline conditions with 1 mol. of salicylic acid, and then in presence of dil. acetic acid with 1 mol. of 1-naphthylamine-6-sulphonic acid. This aminodisazo dye is then diazotised and coupled in presence of Na_2CO_3 with 1 mol. of the monoazo dye aniline-2:5-disulphonic acid→*m*-phenylenediamine to give—



E. S.

Diphenylamine Dyes for Cellulose Esters. Celanese Corpn. of America. *BP* 674,566

Yellow dyes for cellulose acetate, of good wash fastness, are prepared by condensing 1-chloro-2-nitrobenzene-4-sulphonmorpholide with aniline or a *para*-substituted aniline in presence of an acid-binding agent. The products have the formula—



(R = H, Alk, hydroxyalkyl, alkoxy, acylamino, or Ar). Thus, 1-chloro-2-nitrobenzene-4-sulphonmorpholide and aniline are boiled for 3 hr. in suspension in aq. Na_2CO_3.

R. K. F.

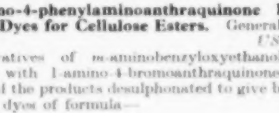
Methylated 1:4:5:8-Tetra-aminoanthraquinone Dye for Cellulose Esters. Celanese Corpn. of America. *USP* 2,562,314

1:4:5:8-Tetra-aminoanthraquinone and its methylated derivative containing minor amounts of the triamino and *β*-substituted derivatives are prepared by nitrating the mixed 1:5- and 1:8-diacetylaminoanthraquinones in 5–6 times their weight of H_2SO_4 containing boric acid. The final product after Na_2S reduction, hydrolysis, and methylation dyes cellulose acetate faster to light and burnt gas fumes than the dye made without boric acid in less H_2SO_4, and does not redden in artificial light.

R. K. F.

1-Amino-4-phenylaminoanthraquinone Derivatives—Dyes for Cellulose Esters. General Aniline. *USP* 2,560,887

Derivatives of *m*-aminobenzyloxyethanol are condensed with 1-amino-4-bromoanthraquinone-2-sulphonic acid and the products desulphonated to give blue cellulose acetate dyes of formula—



(R = H or CH_3; Z = H or hydroxyalkyl; and X = H or $CH_3{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}O{\cdot}Z$) which are fast to burnt gas fumes. Thus, 1-amino-4-bromoanthraquinone-2-sulphonic acid and 2-*m*-aminobenzyloxyethanol are heated at 70°c. for 10 hr. in aq. Na_2CO_3–$NaHCO_3$ containing Cu_2Cl_2. After salting out, the resulting product is desulphonated by treating at 70°c. with aq. ammoniacal sodium hydrosulphite.

R. K. F.

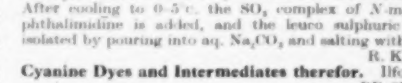
Sulphuric Esters of Leuco Vat Dyes—Use of *N*-Alkylphthalimidines. ICI. *BP* 673,541

Sulphuric esters of leuco vat dyes are made by replacing the amide of *BP* 633,681 (*J.S.D.C.*, **66**, 336 (1950)) by an *N*-alkylphthalimidine (reduce corresponding *N*-alkylphthalimide with Sn and HCl). Thus, 4:5'-dibenzoylamino-1:1'-dianthrimide-2:2'-carbazole is treated at 50°c. for several hours with *N*-methylphthalimidine, methyltriethylammonium chloride, and Zn dust in acetonitrile. After cooling to 0–5°c. the SO_3 complex of *N*-methylphthalimidine is added, and the leuco sulphuric ester isolated by pouring into aq. Na_2CO_3 and salting with KCl.

R. K. F.

Cyanine Dyes and Intermediates therefor. Ilford. *BP* 675,226

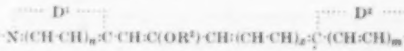
Compounds of formula—



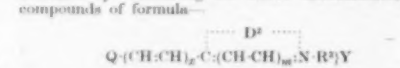
(R^1 = Alk, aralkyl, or Ar; R^2 = Alk or aralkyl; D^1 = residue of a 5- or 6-membered heterocyclic nucleus; n = 0 or 1; X = an acid radical) are of value as dye intermediates, since the terminal CH_3 is very reactive. They are obtained by condensing the appropriate acylmethylene base with an alkyl or aralkyl *p*-toluenesulphonate at < 160°c. to produce compounds where X = *p*-toluenesulphonate and then treating to convert X to another value, e.g. with an inorganic halide.

BP 675,227

Dyes of formula—



(R^3 = Alk, Ar, or aralkyl; D^2 = residue of a 5- or 6-membered heterocyclic nucleus; m = 0 or 1; x = 0 or 1) are obtained by condensing the above intermediates with compounds of formula—

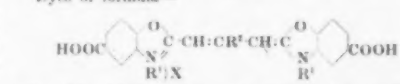


(Y = an acid radical; Q = SR^4 or subst. or unsubst. amino; R^4 = Alk, Ar, or aralkyl).

C. O. C.

Benzoxazole Carbocyanine Dyes. DuP. *BP* 675,360

Dyes of formula—

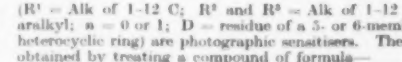


(R^1 = Alk of 1–12 C or benzyl; R^2 = hydrocarbon; X = acid radical) are good non-staining photographic sensitisers.

C. O. C.

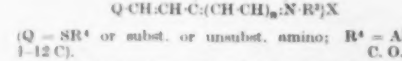
Methin Dyes. Ilford. *BP* 674,003

Dyes of formula—



(R^1 = Alk of 1–12 C; R^3 and R^5 = Alk of 1–12 C or aralkyl; n = 0 or 1; D = residue of a 5- or 6-membered heterocyclic ring) are photographic sensitisers. They are obtained by treating a compound of formula—

with one of formula—

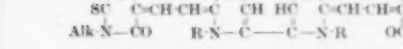


(Q = SR^4 or subst. or unsubst. amino; R^4 = Alk of 1–12 C).

C. O. C.

Symmetrical Tetranuclear Dyes. General Aniline. *USP* 2,586,164

Dyes of formula—



(R = Alk or aralkyl) are non-migrating photographic sensitisers compatible with colour coupling components. C. O. C.

Recovery of Carbon Black from Suspension in a Gas. Phillips Petroleum Co. *USP* 2,585,659

Carbon Black. Phillips Petroleum Co. *USP* 2,587,107

Higher yields of carbon black having increased scorch time and/or lowered pH are obtained by extracting with a chlorinated solvent the tar in the first black obtained and passing some of the solvent to the reaction zone of the carbon black furnace. C. O. C.

Leafing Agent for Bronze Pigment. Reynolds Metals Co. *BP* 674,407

Straight-chain aliphatic amines of > 13 C and mol. wt. 185–300 are not only superior leafing agents, but also mix and disperse more completely and readily in paint and varnish vehicles and result in an improved colour in the leafed pigment. C. O. C.

Fluorescent Zinc Oxide. B. L. Hirsch. *USP* 2,585,461

French-process zinc oxide (i.e. that prepared by burning Zn) of 0·075% moisture content is treated for 1 min. at room temperature with a 100% SO_2 atmosphere, and is then brought to incandescence in a reducing atmosphere. The product may be ground as finely as fluorescent zinc sulphide without affecting its fluorescent properties. C. O. C.

Zinc Sulphide. "Sachtleben" A.-G. für Bergbau und Chemische Industrie. *BP* 674,603

Zinc sulphide of high covering power and tinting strength is obtained by precipitating zinc sulphide and then ageing it in 5–40% H_2SO_4 or 0·1–0·001 N-HCl at 60–100°C. There is no need to calcine or use high pressures. The colouring strength of the product is several times that of lithopone. C. O. C.

Surface-active Agents for Modifying the Surface Characteristics of Organic Pigments and Carbon Black. B. F. Goodrich Co. *BP* 674,832

Organic pigments and carbon black are rendered more readily wettable both by water and by organic liquids by mixing them with a naphthalenesulphonic acid–formaldehyde condensate, a ligninsulphonic acid or ligninsulphonate or a glucoside extract of tannins, and a metal salt whose aq. soln. has pH < 7·0. C. O. C.

Application of Titanium Pigments in Rubber and Plastic Compositions (XIII p. 371).

V—PAINTS; ENAMELS; INKS

Rheology of Carbon-paper Inks. E. S. Gale and B. J. Staneslow. *Amer. Ink Maker*, **28**, (12), 34–41, 67 (1950); *Chem. Abs.*, **46**, 5335 (10 June 1952).

A study of the rheology of carbon-paper inks at 95°C. (temperature at which they are applied to the paper) with a view to the replacement of carnauba by synthetic waxes. A carbon black thought to have a hydrogen rather than a carbon complex adsorbed on it did not disperse more readily in petroleum oils than did a high-colour, low-oil-absorption channel black. A carbon black which normally flowed after treatment with hydrogen had zero flow value. Chlorophyll (freshly prepared from spinach or as the Cu-stabilised oil-soluble commercial form) was one of the best dispersants found but it was considered uneconomical. C. O. C.

PATENTS

Water-emulsifiable Metallic Paste Pigments. Aluminum Co. of America. *USP* 2,587,266–7

Metallic pigments, particularly aluminium flake, produced by either the dry or the paste process, when mixed with mineral oil or other hydrocarbon, palmitic and/or stearic and/or oleic and/or ricinoleic acid, tall oil, a binder, e.g. casein, and a water-soluble soap-forming alkali, or amine, e.g. Na_2CO_3 or ethanolamine, yield a product which is self-emulsifiable in water.

USP 2,587,268

Addition to the paste of one or more anhydrous, water-soluble carbohydrates, e.g. starch, improves the binding and adhesion characteristics of the water paints made from it. C. O. C.

Pigmented Chips. Monsanto. *BP* 674,966

A pigment, a copolymer of a compound of formula R·CH:CH_2 (R = substd. or unsubst. phenyl), a secondary alkyl half-ester of an ethylene-α,β-dicarboxylic acid, and a plasticiser are heated and mixed under shearing stress. The resulting mixture is allowed to harden, and is then broken into chips for colouring lacquers, etc. C. O. C.

Pigmented Coating Compositions. British Thomson-Houston Co. *BP* 674,865

Addition to pigmented resinous coating compositions of 0·000001–0·01% by wt. of the product of hydrolysis of a compound of formula X·$Si(CH_3)_2$·CH_2·$Si(CH_3)_2$·X (X = Cl, Br, or F) decreases the tendency to pigment flotation. C. O. C.

Spirit-soluble Colour for Duplicating Ink. DuP. *USP* 2,586,570

An excellent black is very simply obtained by heating together the bases of Chrysoidine (*C.I.* 20 or 21) (100 parts), Crystal Violet (*C.I.* 681) (66–89), and Victoria Blue (*C.I.* 729) (30–50) and then adding HCl to the melt to form the hydrochlorides. The product is suitable for hectograph inks, as it does not increase the viscosity of the vehicle and its components exhaust at a uniform rate, so that the copies made from it always match. C. O. C.

Oil-free Wrinkle-finish Coating. Union Carbide & Carbon Corpn. *USP* 2,585,197

Wrinkle-finish coatings resistant to alkali are given by the heat-hardenable resins obtained by treating a phenol with a secondary aliphatic amine and condensing the product with formaldehyde. C. O. C.

Unburned Pencil Leads. J. S. Staedtler Mars-Bleistiftfabrik. *BP* 675,321

Unburned pencil leads, lines made by which cannot be mechanically or chemically erased and which are fast to light, are made by mixing lampblack with wetting agents together with binding and filling agents. C. O. C.

Coating Compositions (III p. 358).
Mechanism of Drying Retardation by Toluidine Red Toners (IV p. 362).

VI—FIBRES; YARNS; FABRICS

Review of Textile Research and Development during 1951. Edited by R. Steele and C. Mesaler. *Textile Research J.*, **22**, 293–377 (May 1952).

A review of the literature published during 1951 in the field of textile science and technology, including a bibliography containing 1758 references. A. B.

Birth of the Irish Linen Industry—Articles of Association of the Corporation of the Governour and Assistants of the King and Queens Corporation for the Linnen Manufacture in Ireland. Anon. *British Textiles* (54th ed.), 41–48 (June 1952).

Photographic reproductions of a document dated 16 Nov. 1692, which is in effect the articles of association of the above corporation and authorises it to begin operations in Drogheda, together with a transcription of the text. It is now in the possession of Mr. I. Moore Mitchell of the High Society Linen Co., Belfast. C. O. C.

Delignification of Jute with Sodium Chlorite. H. J. Callow. *J. Textile Inst.*, **43**, T 247–T 249 (May 1952).

Using the Cross and Bevan method of detecting lignin, viz. addition of Na_2SO_3 soln. to a sample of jute after acid chlorination, the effect of repeated treatments with $NaClO_2$ is studied in an attempt to prepare a cellulosic fraction of a purity normally associated with Cross and Bevan or Norman and Jenkins cellulose. Repeated 30-min. treatments with 0·7% soln., near the boil, in pH 5·2 buffer, are each followed by pH 5·5 $NaHSO_3$ extraction. Thirteen treatments still result in a positive colour test, despite 20·8% loss in wt. and 30·8% acetyl loss. Seven treatments in 2·8% $NaClO_2$ remove all traces of lignin, with a wt. loss of 34·1% and acetyl loss of 67·0%. α-Cellulose, xylan, and acetyl contents are very similar to those of Cross and Bevan cellulose. Cuprammonium fluidity shows severe chemical damage, and it is concluded that preparation of a lignin-free fraction from jute must be accompanied by high acetyl loss. J. W. B.

Action of Ketens (Carbon Suboxide and Carbonylmethylene) on Protein, Polyamide, and Cellulose Fibres. H. Rath, H. Meyer, and H. Boerling. *Melliand Textilber.*, **33**, 427–428 (May 1952).

Treatment of wool or of Perlon L with carbon suboxide in toluene leads to a reduction in the affinity of the fibre for acid dyes and to an increase in its affinity for basic dyes. This is considered to result from the blocking of amino groups by the formation of side chains (–NH·CO·CH_2·COOH) and bridges (–NH·CO·CH_2·CO·NH–), and from the presence of anionic groups in the new side chains. It is noted that a short treatment of wool with carbon suboxide leads to increased solubility of the fibre in caustic alkali (preferential formation of anionic side-chains), while longer treatment leads to reduced solubility (increased bridge formation). A polymer of carbon suboxide is formed during treatment and gives the fibre a yellow or brown colour. The polymer can be largely removed by treatment with methanol, dispersing agents, or an oxidising agent ($KMnO_4$ for wool, $NaClO_2$ for Perlon L). When cellulose fibres are similarly treated, their dyeing properties are not greatly affected, but there is some reduction in their tendency to swell. The same materials, also silk and Perlon U, are treated with keten (carbonylmethylene) as a gas or in acetone soln. Treated wool has properties similar to those of wool acetylated with acetic anhydride—increased extensibility, increased tendency to felt, lower swelling, lower affinity for acid dyes. The reserve effect is inferior to that produced by carbon suboxide with respect to its resistance to treatment with hot or acid solutions. Silk and Perlon L, treated with keten, show a similar reserve effect, but Perlon U behaves in the contrary fashion: the untreated fibre is not dyed with acid dyes at pH = 2, but acquires an affinity for such dyes after treatment with keten. Keten-treated cellulose fibres acquire some affinity for acetate-rayon dyes. A. E. S.

Digestion of Wool by Insects. IV—Absorption and Elimination of Metals by Lepidopterous Larvæ, with special reference to the Clothes Moth, *Tineola bisselliella*. V—Goblet Cells in the Midgut of Larvæ of the Clothes Moths and other Lepidoptera. VI—pH and Oxidation Reduction Potential of the Alimentary Canal of the Clothes Moth Larva. D. F. Waterhouse. *Australian J. Sci. Research*, **B 5**, 143–188 (1952); *Chem. Abs.*, **46**, 5210 (10 June 1952).

IV—Moth grubs were fed on food containing 30 metallic and 5 non-metallic elements. 19 elements formed insoluble sulphides in the midgut, where the wool had its S–S bonds reduced to SH groups by alkaline, highly reducing secretions. The alkaline earths were deposited as phosphate granules in the columnar cells of the anterior and posterior midgut. Moth grubs are thus able to detoxify a wide range of metals and non-metals, some highly toxic to other animals.

V—Morphological.

VI—pH and oxidation reduction potentials of the various regions of the gut are given. C. O. C.

Elasticity of Keratin. A. Miyake. *Busseiron Kenkyū (Researches on Chem. Phys.)*, (30), 63–76 (1950); *Chem. Abs.*, **46**, 4889 (10 June 1952).

A statistical-mechanical theory is developed of the elasticity of keratin as treated by grand canonical ensemble. The molecular structure is assumed to be the perfect α-type with no load. Part then changes gradually to β-type by elongation, becoming perfectly β-type with 30% elongation. The effect of water absorption, which occurs on the main chain and affects hydrogen bonds, is taken into consideration by use of the B.E.T. theory of multilayer absorptions. The extension–load curve is compared with Speakman's experimental data. C. O. C.

Statistical Theory of Wool-like Elasticity. A. Miyake. *Busseiron Kenkyū (Researches on Chem. Phys.)*, (47), 79–84 (1952); *Chem. Abs.*, **46**, 4889 (10 June 1952).

A statistical mechanical theory is developed of a general three-dimensional network system of linear high-polymer molecules on the assumption that each kinetic unit (several amino acid residues) of the chain corresponds to the canonical ensemble of the constant stress and that the stresses are in equilibrium at each free junction point of the network. The theory is applied to wool-like elasticity. C. O. C.

Mechanical Properties of Keratin. A. Miyake. *Busseiron Kenkyū (Researches on Chem. Phys.)*, (39), 121–130 (1951); *Chem. Abs.*, **46**, 4889 (10 June 1952).

The theory developed previously (see above) is extended to include general mechanical properties of keratin fibres, with consideration of the mechanism of deformation. The deformation of the molecules consists of the ideal elastic deformation, which is related to that in bulk, and of the deformation due to intermolecular slip; the latter was treated by the theory of rate processes, taking into account the partly remaining network structure of intermolecular binding in slipping. The bulk stress is related to the intermolecular stress. The stress–strain curve under constant rate of load, creep under constant load, and stress relations under constant elongation are derived from the fundamental equations obtained in terms of molecular quantities, and are compared with experimental results obtained by previous workers. C. O. C.

Artificial Fibre in α-Configuration derived from Wool. P. Alexander, D. Carter, C. Earland, and P. F. Mariner. *Melliand Textilber.*, **33**, 187–188 (March 1952).

Wool is converted into an alkali-soluble form by treatment with peracetic acid, which attacks the disulphide linkages without affecting the main chains. A 15% soln. of the product in dil. NH_4 is spun into a soln. containing sodium sulphate, magnesium sulphate, and Fixanol, and the resulting fibre is passed into another soln. containing these same substances and also formaldehyde. The X-ray diagram of the fibre shows it to be in the α-configuration, but poorly oriented. After stretching by 100%, the fibre shows a very sharp α-diagram, and it is evidently stabilised in this form by cross-linkages. A. E. S.

Composition of Silk. F. Happey and A. J. Hyde. *Nature*, **169**, 921 (31 May 1952).

Recent research on the composition of silk is briefly reviewed, and a mechanism for the extrusion of natural silk is suggested. J. W. B.

Configuration of Silk Fibroin dissolved in Lithium Bromide Solution. B. A. Toms and A. Elliott. *Nature*, **169**, 877–878 (24 May 1952).

Concentrated syrups of silk in aq. LiBr are prepared containing 15 and 16% of fibroin, and show a well marked absorption band at 4665 and 4680 cm.$^{-1}$ respectively; a solid film of water-sol. silk shows a band at 4595 cm.$^{-1}$. Thus the silk molecule in solution as well as in soluble solid film is folded, and may be in the α-configuration. J. W. B.

Action of Alkalis and Acids on Fibroin in presence of Neutral Salts. M. V. Korchagin. *J. Appl. Chem. U.S.S.R.*, **25**, 212–216 (Feb. 1952).

The author showed in 1948 that the extent to which fibroin is degraded by treatment at a given pH with carbonate–bicarbonate buffers increases rapidly as the concn. of buffer mixture is increased. These results and the analogous results of experiments now described, in which degummed silk is treated with phosphate and borate buffers, are attributed to a kinetic salt effect. In further experiments, degummed silk is treated with 0·02 N-NaOH for 45 min. at 80°C. in presence of various salts (LiCl, NaCl, NaI, Na_2SO_4, KCl, KBr, KI, $CaCl_2$, $BaCl_2$) at varying concn. (up to 0·4 N.). Similar experiments are done on *raw* silk, using *acid* solutions at 90°C., e.g. 0·03 N-HCl for 60 min. in presence of $CaCl_2$, $BaCl_2$, NaCl, NaBr, NaI, or $NaNO_3$ at a concn. of 0·15 N., and subsequently degumming with soap soln. under standard conditions. The damage produced by these treatments is assessed by measurements of viscosity (in cuprammonium soln., according to the author's method), tensile properties, and flexing resistance. The results confirm the existence of a positive salt effect, which in alkaline soln. is associated with the nature of the cation ($Li^+ < Na^+ < K^+ < Ca^{++}$ and Ba^{++}) and in acid soln. with the nature of the anion ($Cl^- < Br^- < NO_3^-$ and I^-), the effect of the oppositely charged ion being in each case small. The following viscosity figures are selected from the tabulated results to indicate the magnitude of the effects: Treatment with NaOH—without salt, 6·7; with 0·2 N-NaCl, 5·8; with 0·2 N-$CaCl_2$, 3·0. Treatment with HCl—without salt, 0·4; with 0·15 N-NaCl, 4·9; with 0·15 N-NaI, 4·0. A. E. S.

Pilot Plant Production of Peanut Protein. J. Pominski, W. O. Gordon, E. J. McCourtney, H. L. E. Vix, and E. A. Gastrock. *Ind. Eng. Chem.*, **44**, 925–928 (April 1952).

Pilot plant data are presented on the effect of grinding peanut meal before peptisation and on the use of different methods and equipment in separating, dewatering, and drying the spent residue. The data are applicable in counter-current systems of peptisation. The yield of protein was improved appreciably by successive peptisations and also by grinding the meal before peptisation.
W. K. R.

Peanut Protein Fibres—Flow Characteristics of Spinning Solutions as affected by Rate of Extrusion. J. C. Arthur and H. G. Many. *Amer. Dyestuff Rep.*, **41**, 385–386 (23 June 1952).

Apparatus representing a section of the extrusion system for protein fibre shows that, per unit volume of solution delivered, the loss in head diminishes as the rate of extrusion increases. This indicates that the fluidity of spinning solutions of protein fibre is affected by the rate of extrusion. There is a diagram of the apparatus used, and data are given in the form of a graph and a table.
A. H.

Solubility of Cellulose Acetate in Acetone. P. V. Kozlov and F. S. Sherman. *J. Appl. Chem. U.S.S.R.*, **25**, 384–391 (April 1952).

A study is made of the solubility in acetone of a large number of cellulose acetate preparations containing 52–58% of combined acetic acid, and it is concluded that the main solubility-determining factor is the degree of polymerisation (D.P.) of the material. The incomplete solubility of preparations obtained from linters by heterogeneous acetylation and hydrolysis is shown to be due not to the chemical heterogeneity of these products, but to their relatively high D.P. Thus, by separating the sol. and insol. fractions of the material and determining their acetyl numbers and their viscosities in methylene chloride–methanol soln., it is found that the fractions have substantially the same chemical composition, but differ greatly in D.P. The same material after a depolymerisation treatment and material prepared from low-viscosity linters are completely sol. in acetone.
A. E. S.

Mechanical Behaviour of High Polymers. I—Stress–Strain Relationship in Nylon. K. Makashima. *J. Soc. Textile Cellulose Ind.* (Japan), **6**, 328–330 (1950): *Chem. Abs.*, **46**, 4803 (25 May 1952).

Both dry and wet nylon fibres after stretching recovered their original lengths sooner or later, but greater elongation was accompanied by longer hysteresis. The typical sigmoidal stress–strain curves obtained were almost identical for dry and wet nylon, and were approximately the mirror image of a similar curve for wool.
C. O. C.

Morphology of Crystallising Polymers. A. Keller. *Nature*, **169**, 913–914 (31 May 1952).

Examination by polarised light of crystallised polyethylene terephthalate spherulites, and electron micrographs of solvent-cast polyethylene terephthalate films, indicate that during crystallisation the molecules first form fibrils which are flat and helically coiled ribbons. These fibrils aggregate and eventually form spherulites and sometimes larger fibre bundles. Drawn fibres of this polymer are also split into smaller units using suitable solvents, showing ribbonlike microfibrillar units with a tendency to coil into helices. It is suggested that drawing is not simply a molecular process, but that spontaneous fibre formation is inherent in long chain molecules.
J. W. B.

Glass-fibre Investigations. IX—Effects of Water Curing, Heat, and Cold on the Mechanical Strength of Glass Fibres. P. A. Koch. *Glastech. Ber.*, **25**, 44–49 (1952): *Chem. Abs.*, **46**, 4753 (25 May 1952).

The reduction in strength brought about by leaching glass fibres with water is mainly due to removal of sodium from the glass, which is not merely a surface attack. The effect on glass fibres of various makes and deniers is given.
C. O. C.

PATENTS

Obtaining the Wool from Sheepskins. Norman Evans & Rans. *BP* 674,270

Better results are obtained in pieing (removing the wool with the aid of enzymes) if the skin is treated to cause shrinkage of the skin collagen before it is destroyed with the proteases of aerobic spore-forming bacteria, e.g. *Bacillus subtilis*.
C. O. C.

Removing Heavy-metal Salts and other Impurities from Cellulose regenerated from Viscose. Celanese Corpn. of America. *USP* 2,585,141

Treatment of the freshly precipitated cellulose, preferably while in its gel state, with a meta-, tetra-, or polyphosphate removes many impurities which are not removed by ordinary washing, desulphurising, or bleaching.
C. O. C.

Impregnated Cellulose and Cellulose Esters therefrom. DuP. *USP* 2,585,516

Cellulosic materials of greatly increased reactivity to esterification and other reactions are prepared by impregnating cellulosic material with a mixture of an amide or a substd. or unsubst. ammonium carboxylate containing at least one amino H and a compound of an inorganic oxy-acid of sulphur, e.g. H_2SO_4 or H_2SO_3. The impregnated material readily reacts e.g. with aldehydes or anhydrides to form acetals or esters.
C. O. C.

Water-soluble Cellulose Ether Fibres. BrC. *BP* 673,654

A water-soluble methyl cellulose dissolved in aqueous acetic acid is spun into an acetone coagulating bath, and the filaments are stretched to increase their strength.
W. G. C.

Solutions of Polymers or Copolymers of Vinyl Chloride. Vereinigte Glanzstoff-Fabriken. *BP* 674,792

A spinning solution is obtained by dissolving vinyl chloride polymers or copolymers in a mixture of tetrahydrofuran and not > 50% of a solvent for acrylonitrile.
C. O. C.

Nylon of High Safe Ironing Point. DuP. *USP* 2,585,163

Nylon of high safe ironing point and of low shrinkage in boiling water is obtained by treating a mixture of stereoisomers of bis-*p*-amino*cyclo*hexylmethanes (50–90% normally solids) with an equimolecular proportion of glutaric acid under polyamide-forming conditions.
C. O. C.

Viscosity-stable Polyamides of Improved Dyeing Properties. DuP. *USP* 2,585,199

Presence of an *N*-aminoalkylmorpholine during the formation of nylon yields nylon of constant viscosity and of improved affinity for acid dyes. The amount used should be 0·1–5·0 mol.% on the wt. of the diamine component.
C. O. C.

NN-Dimethylacetamide as Solvent for Polymers and Copolymers of Acrylonitrile. Monsanto. *BP* 673,841

NN-Dimethylacetamide is an unusually effective solvent for polymers containing not < 70% of acrylonitrile. Solutions in it are used for making acrylonitrile fibres and have the advantages compared with use of *NN*-dimethylformamide as solvent of enabling higher temperatures to be used, with less loss of solvent and a higher rate of spinning.
C. O. C.

Hexahydro-*s*-triazines as Solvents for Acrylonitrile Polymers and Copolymers. Eastman Kodak Co. *USP* 2,585,242

Polymers and interpolymers containing not < 70% by wt. of acrylonitrile readily dissolve in completely acylated derivatives of a hexahydro-*s*-triazine to clear solutions free from gel particles and with no tendency to gel on standing or exposure to air. On cooling, such solutions crystallise, the solvent then acting as a plasticiser.
C. O. C.

Mixture of Nitromethane and Formamide as Solvent for Acrylonitrile Polymers. American Viscose Corpn. *USP* 2,585,918

Polymers containing not < 85 mol. % of acrylonitrile disperse at room temperature in a mixture of 2–40% by vol. of nitromethane and 98–60% of formamide. When the dispersion is heated the polymer dissolves to yield a homogeneous solution, which is stable at room temperature and which may be used for forming filaments.
C. O. C.

Acrylonitrile Polymers. American Viscose Corpn. *BP* 675,143

The product obtained by treating an acrylonitrile polymer (35–98% acrylonitrile) containing $CO{\cdot}NH_2$ groups with an aqueous alkali-metal hypohalite so as to convert at least some of the carbamyl groups to amino groups gives intensely brilliant dyeings with acid dyes. C. O. C.

Dyed Polyacrylonitrile Fibres. American Cyanamid Co. *BP* 673,738

Dyed fibres of acrylonitrile copolymers are obtained by spinning an aqueous salt solution of the polymer into water at < 10°C. and passing the swollen fibre into a bath containing an acetate rayon dye. W. G. C.

Tris-*NN*-dimethylaminophosphine Oxide as Solvent for Vinylidene Chloride Polymers. Monsanto. *USP* 2,587,464

Tris-*NN*-dimethylaminophosphine oxide (obtained by treating dimethylamine with phosphorus oxychloride) readily dissolves polymers containing not < 85% by wt. of vinylidene chloride at > 50°C. The solution is clear and transparent, and remains liquid indefinitely when cooled. C. O. C.

Acrylonitrile - Vinylidene Chloride Copolymer Fibres. BrC. *BP* 674,323

Acetone-soluble copolymers of acrylonitrile and vinylidene chloride are obtained by keeping the monomer ratios substantially constant during polymerisation, e.g. by bleeding off the vinylidene chloride continuously. They may be used for either wet or dry spinning. W. G. C.

Dye-receptive Acrylonitrile-Amine Oxide Copolymers. Chemstrand Corpn. *USP* 2,586,238

Copolymers of acrylonitrile and compounds of formula $R^1R^2R^3N{\rightarrow}O$ *or* $XR^3N{\rightarrow}O$ (R^1 and R^2 = Alk., Ar, or aralkyl; $R^3 = CH_2{\cdot}CH{\cdot}CH(OR^4){\cdot}CH_2{-}$ or $H{\cdot}[CH_2]_y{\cdot}C(:CH_2){\cdot}CH_2{-}$; R^4 = H or acyl of 1–12 C; x = 0–2; X together with the N of the formula = a "bivalent" radical derived from a heterocyclic secondary amine), e.g. *NN*-dimethylallylamine oxide or *N*-allylmorpholine oxide, are soluble in a wide range of solvents and are readily dyed with acid dyes. C. O. C.

Heterocyclic Polymers. M. M. Sawarc. *BP* 673,652

Solid polymers are obtained by cooling the vapour produced by heating at 700–1000°C. for < 10 sec. an aromatic heterocyclic compound having only C and N in the six-membered ring and having two CH_3 groups *para* to one another, e.g. 2:5-dimethylpyridine. They can be spun from acid solution into an alkaline bath to form filaments. W. G. C.

Review of Dyes and Machinery suitable for Felts and Galloons (I p. 356).
Use of Rare Oxides for Colouring and Decolorising Glass (VIII p. 369).
Dyeing Viscose, Cellulose Esters and Ethers, Polyamides, Polyurethans, Polyesters, Polyacrylonitrile, and Polyvinyl Chloride (VIII p. 369).
Basic Degrese of Polymerisation of Cellulose Acetate (XIV p. 372).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Nature of Stabiliser present in Sericin. V. N. Krestinskaya and M. B. Aymukhamedova. *J. Appl. Chem. U.S.S.R.*, **25**, 197–204 (Feb. 1952).

Further work on solutions containing sericin and its hydrolysis products (cf. J.S.D.C., **68**, 279 (July 1952)) indicates that the substances present that are active in peptising sericin and stabilising it in colloidal soln. are not simply peptides, but are peptides that have adsorbed amino acids. It is confirmed that simple amino acids alone are inactive, but it is shown that the activity of cocoon extracts is increased by addition of amino acids, while at the same time the amount of amino-acid nitrogen revealed by analysis becomes less than the total amount present in the solutions before mixing. A. E. S.

Variation of Electrical Conductance with Temperature of Aqueous Sodium Oleate Solution in presence of Different Alcohols and Salt. A. N. Bose. *J. Indian Chem. Soc.*, **29**, 135–139 (Feb. 1952).

The effects of n-propyl, isopropyl, n-butyl, and n-amyl alcohols in presence of NaCl on the specific conductance of soap solution have been studied. Specific conductance plotted against temp. shows that at lower concentrations of alcohol straight-line plots are obtained, but at higher concentrations the plots are concave upwards. This is probably due to the fact that at low dilutions of alcohol the number of alcohol molecules in the palisadal layer is small, and hence the size and charge of the micelle do not appreciably change with temp. At higher concentrations of alcohol the palisadal layers are saturated with alcohol molecules, and change of temp. increases the size of the micelle and diminishes the charge. Addition of NaCl does not appreciably change the nature of the curves. n-Propyl and isopropyl alcohols show a more marked curvature in plot than the other two, probably owing to hydration of the molecules. A. H.

Soiling and Soil Retention in Textile Fibres. W. J. Hart and J. Compton. *Ind. Eng. Chem.*, **44**, 1135–1141 (May 1952).

The kinetics of the deposition of grease-free carbon black on to chopped cotton fibres has been studied, and the effects of concentration of the soil, concentration of the fibre, intensity of agitation during soiling, particle size of the soil, presence of surface-active agents, and temperature have been investigated. The results confirm the view that soiling is due to micro-occlusion rather than to sorption (cf. Compton and Hart, *Ind. Eng. Chem.*, **43**, 1564 (1951)—J.S.D.C., **67**, 475 (1951)). The factors controlling the formation and stability of the fibre–soil complex are considered to be the geometric relationship between the sizes and shapes of the soil particles and the sizes and shapes of the cavities in the cotton fibre surface, and the probability of close approach. W. K. R.

Carbonising by Radio-wave Heating. I—Some Properties of Wool in High-frequency Electric Fields. N. Azuma, E. Kuze, and F. Bekku. *J. Soc. Textile Cellulose Ind.* (Japan), **6**, 374–376 (1950).

II—Carbonising Burrs in Felt. N. Azuma, I. Yamamoto, F. Bekku, F. Yamashita, and Y. Niwa. *Ibid.*, 437–440; *Chem. Abs.*, **46**, 4804 (25 May 1952).

I—The dielectric constant and power factor tan δ of wool in high-frequency waves tended to increase (hence generate more heat) with large apparent sp. gr. when the water content was constant and with larger water content when the apparent sp. gr. was constant.

II—Burrs in wool carbonised quicker and with less damage to the wool on being heated with 10-megacycle waves than with ordinary steam heating. C. O. C.

PATENTS

Bleaching Wood Pulp. Canadian International Paper Co. *USP* 2,587,064

When chemical pulps are bleached first with chlorine dioxide and then with a small amount of hypochlorite, the brightness, whiteness, and colour stability are higher than can be obtained with either bleaching agent alone or with hypochlorite followed by chlorine dioxide. Degradation is much less than when a hypochlorite is used to develop the maximum brightness it will produce. C. O. C.

Cleaned and Degreased Slubbing. Bigelow-Sanford Carpet Co. *USP* 2,587,504–5

Scoured wool is sprayed during picking with an aqueous dispersion of bentonite so as to deposit 1–2% bentonite in it. The bentonite absorbs grease and dirt left in the wool, and most of it together with the attached grease and dirt falls out during carding, thus giving slubbing which contains only half the dirt, etc. it otherwise would. 0·25–1·0% Bentonite remains in the wool, and prevents it from picking up dirt, giving a yarn which when woven into carpets is readily cleaned by household vacuum cleaners. C. O. C.

Use of Cathodic Polarisation for Preventing Corrosion in the Textile Industry (I p. 356).
Organic Complexing Agents in Textile Processing (II p. 357).
Chain Mechanism of Decomposition of Hydrogen Peroxide and Existence of the Radical HO_3 and of A. N. Bakh's Higher Peroxide of Hydrogen (III p. 357).
Action of Alkalis and Acids on Fibroin in presence of Neutral Salts (VI p. 366).
Measurement of Fabric Soiling (XIV p. 372).

VIII—DYEING

Hat Dyeing. G. Kittan. *Textil-Praxis*, **7**, 224–227 (March 1952).

A practical review of hat dyeing, with special reference to the use of the paddle, cone, and Obermaier machines. B. K.

Effect of Temperature on the Sorption of Orange II by Wool. B. Benson, J. Kruger, R. Donovan, and P. Larose. *Canadian J. Technol.*, **30**, 105–115 (May 1952).

The sorption of Orange II from acid soln. by wool is shown to increase as the temperature is lowered. The effect is quite large and has not been remarked upon by previous investigators. An equation representing the relationship between the amount sorbed at equilibrium is found to be of the same form at 60°c. as that previously reported for 100°c. The energy changes accompanying the sorption of Orange II are calculated using the Gilbert–Rideal equation. A. H.

Marhen Process for Vat-dyebath Control. D. E. Marnon. *Amer. Dyestuff Rep.*, **41**, P 292–P 296 (12 May 1952).

Electrical connection is made to an alkaline-reducing vat dye solution both through a bright Pt electrode and through a saturated calomel–KCl electrode of fixed potential. The potential difference between the two electrodes, aided by a high-input impedance amplifier, which protects the Pt electrode from polarisation, enables the reducing condition of the bath to be measured under commercial dyeing conditions. The process is applied to Williams unit–water-phase dyeing, pad–steam dyeing, and hot-oil dyeing continuous processes and batch jig-dyeing, with excellent control and saving of chemicals. J. W. B.

Theoretical and Practical Studies of Gas Fading. V. S. Salvin, W. D. Paist, and W. J. Myles. *Amer. Dyestuff Rep.*, **41**, P 297–P 304 (12 May 1952).

Gas-fume fading, its mechanism in relation to the fabric, and the use of newer dyes and inhibitors are discussed. Data are presented on the absorption of NO_2 by cotton and nylon, and it is found that acetyl value of the fibres, as well as moisture content, influences the rate of fading. An attempt is made to correlate structures and properties of inhibitors. 46 papers and 75 patents are listed in the bibliography. J. W. B.

Dyeing Polyamide Fibres with Indigosols. C. T. Speiser. *Amer. Dyestuff Rep.*, **41**, 349–350 (26 May 1952).

Dyeings on nylon of good fastness to light are obtained with Indigosols and vat dyes if salicylic acid (25 g./litre) is incorporated in the developing bath. The treatment usually produces a slightly duller and weaker shade. Further treatments to improve light fastness, e.g. steaming, are unnecessary. W. K. R.

Chemicals in Food—Legal and Administrative Background to Problems. C. A. Adams. *Chem. and Ind.*, 484–487 (31 May 1952).

Use of Rare Oxides for Colouring and Decolorising Glass. O. R. Herfurth. *Glas–Email–Keramo–Tech.*, **3**, 1–4, 38–40 (1952).

PATENTS

Dyeing Skins and Furs with Insoluble Azo Dyes. NCO. *BP* 674,638

The goods are treated, before or after dressing and killing, with a strongly caustic alkaline solution of a coupling component having at least one OH group and capable of coupling in *ortho* position to that group. Treatment is carried out in presence of H·CHO as a protecting agent for the goods. Finally the impregnated goods are treated with a diazonium compound. Deep dyeings may be obtained and the dyed hairs retain their natural gloss. The leather is light-coloured, as penetration of the dye into the leather is slight, and the undyed leather is exposed on subsequent shaving of the leather. C. O. C.

Dyeing Viscose, Cellulose Esters and Ethers, Polyamides, Polyurethans, Polyesters, Polyacrylonitrile, and Polyvinyl Chloride. Basf. *BP* 675,244

Very fast dyeings are obtained on the above fibre-forming substances with water-insoluble esters of anthraquinone leuco vat dyes and monocarboxylic acids of < 8 C; e.g. addition of *leuco*-dibenzanthrone acetate to a viscose spinning solution results in deep red-brown filaments of excellent fastness to light and washing. C. O. C.

Colouring Glass Fibres. Owens-Corning Fiberglas Corpn. *USP* 2,582,919

Borosilicate glass fibres containing no alkali-metal oxides are leached, e.g. with aq. HCl, until they are sufficiently porous to absorb dyes. They can then be dyed with basic dyes or treated with a basic mordant, e.g. hydrated aluminium oxide, followed by an acid dye; or an organic pigment, e.g. Lithol-red 2B, may be applied at the same time as the mordant. C. O. C.

Review of Dyes and Machinery suitable for Felts and Galloons (I p. 356).

Ultrasonics in the Textile Industry (I p. 356).

Organic Complexing Agents in Textile Processing (II p. 357).

Increasing the Fastness to Washing of Direct Dyes (III p. 357).

Addition Reactions of Quinones. I—Reaction of Cysteine and Thiourea and its Derivatives with some Quinones (IV p. 361).

IX—PRINTING

PATENTS

Patterned Transparent Cellulose Fabrics. Radmer & Co. *BP* 673,182

A paste containing a natural or synthetic rubber latex as the binder together with vulcanisation, thickening, stabilising, stiffening, and if desired colouring agents is printed on, and then vulcanised to impart a resist to subsequent transparentising treatment. The effects obtained resist washing. C. O. C.

Producing Coloured Design in Hosiery or other Non-woven Textile Fabrics. May Hosiery Mills. *USP* 2,587,534

A machine for screen printing socks, etc. is described. Pretreatment of the goods with an aqueous emulsion of a wax or oil stops the paste from flowing beyond the edges of the design and so results in designs having sharp edges. C. O. C.

Colour Printing by the Four-colour Method. IC. *BP* 674,025

An improved method of making corrected colour-separation images. C. O. C.

Electrostatic Printing. Battelle Development Corpn. *BP* 675,398

X—SIZING AND FINISHING

Digestion of Wool Keratin by Papain–Bisulphite–Urea and Related Systems. F. G. Lennox. *Australian J. Sci. Research*, **B 5**, 189–209 (1952); *Chem. Abs.*, **46**, 5322 (10 June 1952).

Addition of urea greatly assisted digestion of wool in papain–bisulphite solutions, reaching completion in a few hours at 50–70°c. at the optimum pH 7. Incomplete digestion occurred in urea–bisulphite solutions containing ficin or bromelin at pH 7, or pepsin at pH 3; mould protease, trypsin, beef liver cathepsin, and pig pancreas extract showed little activity at pH 5·7–9·6. Digestion was less when $NaHSO_3$ was replaced by other S-containing reducing agents. Thiourea was more effective than urea; guanidine was slightly less effective than urea; while ethylurea, methylurea, urethan, formamide, acetamide, and acetamidine were much less effective in that order. C. O. C.

PATENTS

Rendering Cellulosic Materials Shrink-resisting. Cluett, Peabody & Co. *BP* 673,642

Regenerated or natural cellulosic materials are rendered shrink-resisting by treatment with an aqueous solution of a formaldehyde–formamide (3 : 2–6 mol.) precondensate in presence of an acid catalyst of, or giving rise to an acid of, dissociation constant not $< 1{\cdot}76 \times 10^{-4}$ at 25°c., e.g. $AlCl_3$, the pH of the solution being not > 5, followed by the usual baking. The treated material does not smell of formaldehyde on heating and/or storing. C. O. C.

Relaxation-shrinkage of Tubular Knitted Fabrics. F. R. Redman. *BP* 673,088

The residual shrinkage of tubular knitted fabric can be brought to < 2% by passing it over laterally spaced expanders, which expand it widthwise internally and shorten it in length, sufficient lengthwise freedom being allowed to permit the widthwise expansion without imparting appreciable lengthwise tension. C. O. C.

Imparting Crease- and Dye-resistance to Partly Carboxymethylated or Phosphorylated Cellulosic Fibres. U.S. Secretary of Agriculture. *USP* 2,584,114

Phosphorylated or partly carboxymethylated cotton cloth, e.g. that prepared according to *USP* 2,448,153 (*J.S.D.C.*, **65**, 316 (1949)), is impregnated in the acid state with a solution of a precondensate of the urea- or melamine-formaldehyde type in absence of a catalyst, dried at < 110° C., and baked at 110–180° C. The product is crease-resistant and has no affinity for many direct cotton dyes. C. O. C.

Acrolein and Sulphur Halide for Strengthening Protein Fibres. U.S. Secretary of Agriculture. *USP* 2,583,574

Natural or artificial protein fibres are strengthened, presumably by cross-linking, if they are first treated with a 20% sat. soln. of acrolein at 4–120° C. and then with a sulphur halide in an inert organic solvent at 20–120° C. C. O. C.

Heat-setting of Polyethylene Fabrics. American Viscose Corpn. *BP* 673,879

Fabrics made from "annealed" polyethylene monofilaments or yarns are heated to between their initial shrinkage temperature and their m.p. while in open width and in absence of tension. C. O. C.

Flame-resistant Finish. American Cyanamid Co. *USP* 2,582,961

Textiles of all types are rendered permanently resistant to flaming, creasing, and shrinking with much less loss in tensile strength by impregnation with an aqueous solution containing a thermosetting urea- or melamine-formaldehyde condensate (2–20 parts by wt.), a hydroxymethyldicyandiamide (10–35), and an oxygen-containing acid of phosphorus (equivalent to 5–30 parts of 85% H_3PO_4) followed by drying and then baking at 250–400° F. C. O. C.

Water-repellent Rot-resistant Textiles. Socony-Vacuum Oil Co. *USP* 2,582,870

The material is first impregnated with a water-soluble salt of Cu, Zn, Hg, or similar metal, which is then converted into a water-insoluble salt *in situ*. Finally it is impregnated with an aqueous solution of a derivative of a nitrogenous resinous material containing at least one 5-membered heterocyclic radical and preferably at least one thiophen radical per polymer unit as described in *USP* 2,559,260 (*J.S.D.C.*, **68**, 137 (April 1952)). C. O. C.

Giving Textiles and/or Rubber Resistance to Microbial Attack. Dunlop Rubber Co. *BP* 675,200

A metal salt of 2,2′-dihydroxy-5,5′-dichlorodiphenylmethane, having solubility in water at 25° C. of < 0·20 part by wt. of solution, is incorporated in the material. C. O. C.

Action of Ketens (Carbon Suboxide and Carbonylmethylene) on Protein, Polyamide, and Cellulose Fibres (VI p. 366).

Cleaned and Degreased Slubbing (VII p. 368).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

PATENTS

Felt-like Fibrous Products. American Viscose Corpn. *BP* 674,577

Coherent, strong, highly porous and absorbent felt-like fibrous articles, characterised by high wet strength, are made of randomly intermingled discontinuous fibres, some of which are unbeaten non-fibrillating fibres $\frac{1}{32}$–1 in. long bonded at their points of intersection by a water-insoluble, alkali soluble cellulose ether present in the article in discontinuous, particulate condition through precipitation. Most or all of the non-fibrillating fibres, which may be regenerated cellulose, may by $\frac{1}{4}$–1 in. long, and not < 10% of the non-fibrillating fibre may be $\frac{1}{32}$–$\frac{1}{16}$ in. long, the other fibres present being natural, papermaking fibres. The aqueous dispersion of artificial fibres and the cellulose ether may be diluted with water, or the felt-like article may be partly dried, before precipitation of the ether with sulphuric acid. The ether may be hydroxyethyl cellulose, and the aqueous medium may contain formaldehyde and have an acid pH. S. V. S.

Coating Compositions for Paper. Monsanto. *BP* 674,876

A coating composition for paper comprises an aqueous solution of (i) a copolymer or a water-soluble (e.g. ammonium) salt of a copolymer of a polymerisable vinyl compound of formula $R{\cdot}CH{:}CH_2$ (R = subst. or unsubst. phenyl) and an alkyl half-ester of an ethylene-$\alpha\beta$-dicarboxylic acid and/or the free acid or its anhydride, and (ii) a subst. or unsubst. unsat. fatty acid of > 11 C and/or a water-soluble salt thereof. The copolymer may contain 1·1–5 mol. of the vinyl compound (e.g. styrene) and the reaction product of 1·0–0·4 mol. of a monohydric aliphatic alcohol and 1 mol. of the ethylene-$\alpha\beta$-dicarboxylic acid or anhydride, e.g. maleic acid. The unsaturated fatty acid or salt may contain 15–18 C, e.g. oleic, ricinoleic, or linoleic acid or their ammonium salts, and may be present to the extent of 10–100% of the weight of the copolymer or its salt. A dye, lake, or pigment may be present, the copolymer or its salt amounting to 5–30% of the wt. of the pigment. The coating is applied to at least one surface of the paper base stock, dried, and calendered, yielding an improved coated paper, with good water-resistance, embossable without chipping or cracking, and having a smooth, level, uniform, and non-rubbing surface particularly suitable for fine half-tone printing. S. V. S.

Porous Foils from Viscose as Leather Substitute. Vereinigte Glanzstoff-Fabriken. *BP* 674,203

Solubility of Cellulose Acetate in Acetone (VI, p. 367).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Effect of Anionic Detergents on Collagen. K. H. Gustavson. *Acta Chem. Scand.*, **4**, 1171–1184 (1950): *Chem. Abs.*, **46**, 5350 (10 June 1952).

Data on fixation of Cr by collagen are used to show that pretreatment of collagen with 10% aq. Na dodecyl sulphate causes permanent changes in the collagen involving the co-ordinate rather than the electrovalent bonds. C. O. C.

Anhydrides of *N*-Acylaspartic and *N*-Acylglutamic Acids. J. M. Swan. *Nature*, **169**, 826–828 (17 May 1952).

N-Acylaspartic and *N*-acylglutamic acid anhydrides possess the true anhydride rather than the oxazolone structure. J. W. B.

α-Helix Structure of Poly-γ-methyl-L-glutamate. H. L. Yakel, L. Pauling, and R. B. Corey. *Nature*, **169**, 920 (31 May 1952).

Further evidence is given in support of the 18-residue 5-turn α-helix configuration for poly-γ-methyl-L-glutamate. J. W. B.

PATENTS

Ultrasonics in the Wet Treatment of Leather and Fur. F. Zapf. *BP* 675,237

Application to the processing liquor of frequencies of 200,000–3,000,000 cycles/sec. greatly reduces the period of time needed for processing and results in more intensely tanned, dyed, or greased goods. C. O. C.

Drying Leather. United Shoe Machinery Corpn. *USP* 2,586,985

A rigid drying board coated with chlorinated rubber or neoprene is given a further dried coating of a water-sensitive polymeric amide. Wet leather is slicked out on the board using methyl cellulose paste as the adhesive. The leather is then dried by passing a current of air over it, after which it is readily stripped clean from the board. C. O. C.

Drying Hides and Furs. Frick Co. *USP* 2,587,485

The goods after tanning or dyeing are placed on frames, frozen, and then while frozen treated with air having a

dew-point lower than the temperature of the goods until they are dried by sublimation of the ice. This is a cheap method of drying. The ice formed in the goods breaks down their cellular structure and so yields a soft, pliable, leather or fur of superior quality. C. O. C.

Artificial Fibre in α-Configuration derived from Wool (VI p. 366).

XIII—RUBBER; RESINS; PLASTICS

Application of Titanium Pigments in Rubber and Plastic Compositions. J. Breckley. *Rubber Age* (N.Y.), **70**, 597–605 (1952): *Chem. Abs.*, **46**, 5352 (10 June 1952).

The history, chemistry, and manufacture of TiO_2 are reviewed. Rutile pigments have greater tinting and opacifying power than the anatase, but the latter are the whiter. Vulcanised rubber and plastics withstand sunlight and weather most successfully when they are pigmented with rutile TiO_2, particularly the chalk-resistant grades. The most stable aqueous dispersions of TiO_2 are obtained with those pigments which disperse only with the aid of a dispersing agent. C. O. C.

Possibility of Photosensitised Polymerisation of Styrene with Organic Dyes by Virtue of its Triplet State. M. Koizumi, Z. Kuroda, and A. Watanabe. *J. Inst. Polytech. Osaka City Univ.*, Ser. C, **2**, (1), 1–4 (1951): *Chem. Abs.*, **46**, 4915 (10 June 1952).

The triplet state, or phosphorescence, associated with certain dyes is due to diradicals. These compounds, when activated, may induce a free-radical reaction, e.g. polymerisation. Comparison of the degree of polymerisation of styrene in presence and absence of the activating compound and in the light and the dark indicated that the effect does occur and is activated by light. The dyes used were trypaflavin (I), fluorescein, eosin, Rhodamine 6G, Illuminol R, Illuminol RII (II), Illuminol U (III), and Zn tetraphenylchlorine (IV). (I), (II), and (III) showed the greatest effect. (IV) showed an inhibiting action, although its triplet state is long-lived. The effect is greatly dependent on temperature. C. O. C.

Light Stability of Polystyrene and Polyvinylidene Chloride. L. A. Matheson and R. F. Boyer. *Ind. Eng. Chem.*, **44**, 867–874 (April 1952).

The causes of yellowing and loss of strength of polystyrene and polyvinylidene chloride on exposure to light have been investigated. Polystyrene is subject to photochemically activated oxidation, which is very sensitive to impurities present in the polymer, particularly styrene monomer and sulphur compounds. Yellowing is inhibited by the use of amines of ionisation constant $> 10^{-7}$ as antioxidants. Polyvinylidene chloride is little affected by oxygen, and yellowing is due to loss of HCl, which may be caused by ultraviolet absorption, heat, and chemical action, resulting in the formation of conjugated unsaturation. Discoloration and degradation may be controlled by the use of a stabiliser to absorb the HCl (e.g. phenoxypropylene oxide) or to react with the resulting unsaturation (e.g. esters of maleic acid), by the use of an ultraviolet absorbent (e.g. 2:2′-dihydroxybenzophenone), or by a combination of these types. W. K. R.

PATENTS

Stabiliser for Synthetic Rubber. United States Rubber Co. *USP* 2,587,477

Alkyl phosphates which contain 1–4 quinquevalent P atoms and in which at least one H atom of the acid is unsubstituted, e.g. *di*isoamyl acid pyrophosphate, are excellent stabilisers for synthetic rubber. C. O. C.

Alkylthiosulphenyl Dithiocarbamates as Vulcanisation Accelerators. Phillips Petroleum Co. *USP* 2,586,769

Compounds of formula $R^1{\cdot}NR^2{\cdot}CS{\cdot}S{\cdot}S{\cdot}S{\cdot}Alk$ (R^1 and R^2 = H, hydrocarbon radicals, or radicals which form a heterocyclic ring with the N, but only one can be H) are highly efficient vulcanisation accelerators. C. O. C.

Activators for Vulcanisation Accelerators. Monsanto. *BP* 674,472

Use in addition to a primary organic vulcanisation accelerator of a compound of formula $R^1{\cdot}SO_2{\cdot}NH{\cdot}R^2$ (R^1 = an aromatic group; R^2 = H or Alk) results in more even vulcanisation. C. O. C.

Resinous Compositions. American Cyanamid Co. *USP* 2,585,827

The resins obtained by treating a polyhydric alcohol with a perfluorinated saturated aliphatic polycarboxylic acid or anhydride, e.g. tetrafluorosuccinic acid, have better colour and are more stable to ageing and heat than those prepared from the corresponding chloro, bromo, or iodo acids. C. O. C.

Heat and Light Stabiliser for Vinyl Resins. G. J. Chertoff. *BP* 674,164

Stannous salts of organic acids are more effective stabilisers than the stannic compounds heretofore proposed. C. O. C.

Blending Extrudable Thermoplastic Material with Pigments and other Modifying Agents. Dow Chemical Co. *BP* 674,716

A small amount of the powdered or granular thermoplastic is mixed with 25–50 times the amount of pigment or modifier required in the final product. It is then formed into pellets, which are tumbled and mixed. This concentrated premix is then used to colour or otherwise modify a larger amount of the thermoplastic, a procedure being described by which a uniformly coloured or modified thermoplastic product can be produced continuously. The method is particularly applicable to polystyrene. C. O. C.

Polymerised Methyl Vinyl Ketone Latices (III p. 358).
Vulcanisation Agents (III p. 358).
Pigment Colours for Plasticised Vinyl Chloride Polymers (IV p. 362).
Impregnated Cellulose and Cellulose Esters therefrom (VI p. 367).
Giving Textiles and/or Rubber Resistance to Microbial Attack (X p. 370).

XIV—ANALYSIS; TESTING; APPARATUS

Accuracy of Visual Colour Measurements on Dyed Silk. H. Goeroldt, M. Richter, and H. Weise. *Melliand Textilber.*, **33**, 335–338 (April); 454–457 (May 1952).

The colour of each of 27 dyeings on silk is examined by four different methods, viz. by photospectrometric measurements, by means of the Guild–Bechstein tricolorimeter, by means of the Leifo colorimeter, and by the Ostwald method, using a Pulfrich photometer. In each case the results are expressed in terms of trichromatic co-ordinates and represented as points on a C.I.E. diagram. The spectrometric values are taken as standard, and the orientation of the values obtained by the other methods with respect to the MacAdam ellipse is examined. It is shown that the trichromatic instruments are not so sensitive for matching as the unaided eye. The Ostwald method is even less satisfactory. The limited accuracy of the tricolorimeters is attributed to the considerable differences that occur in spectral character between the light from the pattern and the mixed light beams with which it is matched. Under these circumstances, observation must be limited to a field of foveal magnitude, i.e. $\sim 2^\circ$ instead of, say, 18° in normal visual examination. The possibility of developing more sensitive instruments is discussed. A. E. S.

Constant Temperature and Humidity Chamber. F. Hill. *J. Textile Inst.*, **43**, P 252–P 253 (May 1952).

A simple controlled humidity room is described, with notes on factors influencing efficiency. J. W. B.

Testing of Water-repellent Properties of Raincoatings. H. Arnold. *Melliand Textilber.*, **33**, 322–323 (April); 429–430 (May 1952).

The more important methods available for testing the water-repellency of raincoating fabrics are surveyed, and an American method (*A.A.T.C.C. Yearbook*, 261 (1942)), in which the weighed dry material is immersed in water, squeezed, after sandwiching between filter papers, through a mangle, and reweighed, is recommended for wide use on account of the simplicity of the apparatus required and the relative consistency of the results. Data obtained by this method are given in illustration of the reproducibility of

the results and of the high proofing efficiency and laundering resistance of a Rotal WF finish, as compared with two other unnamed finishes that are in general use. A. E. S.

Leakage through Ground Glass Joints in Alkoxy Determinations. R. G. Stuart. *Chem. and Ind.*, 520 (7 June 1952).

Low results in alkoxy determinations can be caused by adsorption of alkyl iodides both on rubber and on glass-joint lubricants. This can be avoided by an all glass apparatus incorporating an excess-CO_2 outlet through a secondary bubble counter between the CO_2 source and the primary bubble counter. The main gas current is aspirated through the primary counter, the reaction flask and the spiral absorber containing bromine solution. Rates of flow are easily controlled, and, as the joints are under reduced pressure, any leak is inwards. J. W. B.

Light Fastness Standards for Polyvinyl Chloride. E. J. G. Stanley. *J. Oil & Col. Chem. Assocn.*, **35**, 41 (Jan. 1952).

The British Plastics Federation has submitted a draft specification for polyvinyl chloride film and sheet to the British Standards Institution which will shortly be published as a British Standard. From tests carried out at a number of places the conclusion has been reached that the eight blue wool standards of *B.S.*1006 form a satisfactory basis for assessment of fading. A. H.

Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. X—Titration of Small Amounts of Gallium. R. Belcher, A. J. Nutten, and W. I. Stephen. *J.C.S.*, 2438–2439 (June 1952).

Surface-active Agents for Detergent Applications. A. M. Mankowich. *Ind. Eng. Chem.*, **44**, 1151–1159 (May 1952).

It is proposed to select detergents for specific applications by establishing numerical criteria for the fundamental or prime factors in the washing process, and by classifying soils with reference to these numerical values of the prime factors, thereby obtaining the minimum numerical criteria for each prime factor necessary to achieve soil removal in specific soil–surface combinations. Suspending power (*a*) and micellar solubilisation (*b*) are considered to be the prime factors. Procedures are described for obtaining numerical values for (*a*) by measuring the suspendability of manganese dioxide and of carbon black, and for (*b*) by measuring the extent of solubilisation of a water-insoluble dye, e.g. Orange OT. Tables show the ratings, based on these tests, of a wide range of anionic and non-ionic detergents of different chemical types under various conditions of concentration, temperature, and pH, and in the presence of builders. W. K. R.

Polarography of Picric Acid. P. E. Wenger, D. Monnier, and H. Schmidgall. *Helv. Chim. Acta*, **35**, 1108–1114 (June 1952).

It is found that only an alkaline solution at pH 12 gives satisfactory results for the polarographic estimation of picric acid. H. H. H.

Some Applications of Electronics in the Pigment and Related Industries. J. T. Richmond and P. E. Watson. *J. Oil & Col. Chem. Assocn.*, **35**, 162–177 (April 1952).

A brief account is given with simple circuit diagrams of electronic devices for estimating e.g. metal radicals by polarography, viscosity of opaque solutions, degree of glossiness, and the ratio of rutile to anatase in titanium dioxide by X-rays. The uses of radioactive tracer elements, photoelectric cells, and counter and oscillating circuits are also reviewed. C. H. R.

Molecular Weight of Jute α-Cellulose. K. B. Pal, H. Chatterjee, and P. B. Sarkar. *Nature*, **169**, 845–846 (17 May 1952).

α-Cellulose is isolated by NaOH treatment on jute delignified by Cross and Bevan, $NaClO_2$, Norman and Jenkins, and ClO_2 methods, and the mol. wt. determined from cuprammonium fluidities. The value decreases in the above order, indicating some degradation (greatest with ClO_2) during isolation. Use of 9·3% instead of 17·5% NaOH also gives a higher value for the mol. wt. J. W. B.

Basic Degree of Polymerisation of Cellulose Acetate. L. A. Cox and O. A. Battista. *Ind. Eng. Chem.*, **44**, 893–896 (April 1952).

A procedure is presented for measuring the basic degree of polymerisation of cellulose acetates, irrespective of acetyl content, by complete deacetylation and simultaneous solution in cuprammonium hydroxide. The method has wider applicability than measuring the viscosity in organic solvents, and is particularly suitable for directly comparing the basic degree of polymerisation of acetates of differing acetyl content. Viscosity data for cellulose acetates in various solvents are compared with the results obtained for the corresponding regenerated cellulose in cuprammonium hydroxide, and a curve is given for converting the viscosity of acetates of 38–41% acetyl content, measured in acetone–water (96 : 4), into basic degree of polymerisation based on the cuprammonium procedure. W. K. R.

Measurement of Fabric Soiling. A.A.T.C.C., New York Section. *Amer. Dyestuff Rep.*, **41**, P 322–P 340 (26 May); P 389 (23 June 1952).

It is considered that textiles may become soiled by (*a*) diffusion of very small particles from the air, (*b*) deposition of medium and large particles, (*c*) direct transfer from a soiled surface, (*d*) interception of particles in an air stream, (*e*) contact by inertia effect with particles in a moving air stream, and (*f*) electrostatic attraction. The soil may be retained by mechanical occlusion, oil bonding, and electrostatic forces. It is thought, however, that electrostatic effects are of minor importance. Three methods are described for testing the susceptibility to soiling of fabrics and finishes. In each the result is based on a control tested under identical conditions. (1) *Blower Test*—A stream of air is blown through the pattern under controlled conditions for a given time, and soiling is measured by change in reflectance. The results are sensitive to fabric construction and also differentiate finishes on the same fabric. (2) *Tumbler Test*—Patterns are tumbled for 15 min. in a 1-pint jar containing fifty ¼-in. steel balls and 5 g. soil (1 bone charcoal, 10 sodium sulphate) in a Launderometer-type apparatus, and then reshaken to remove excess soil. Soiling is measured by change in reflectance and in sulphate content. This test is less sensitive to fabric construction, but is useful for evaluating the soiling tendencies of different fibres and finishes. (3) *Floor Test*—Patterns are inserted into holes cut in a small rug, which is then subjected to normal service for sufficient time for the control to become reasonably soiled as measured by change in reflectance. This test produces soiling principally by deposition and transfer. It demonstrates differences in the susceptibility to soiling of different fibres and finishes. W. K. R.

Further Tables for Use in computing Small Colour Differences. G. L. Buc. *Amer. Dyestuff Rep.*, **41**, 353–355 (9 June 1952).

ΔV_g, $\Delta(V_g - V_y)$, and $\Delta(V_g - V_y)$ are computed from C.I.E. tristimulus values X, Y, and Z by means of Nickerson's tables. $(0{\cdot}23\ \Delta V_g)^2$, $\Delta(V_g - V_y)^2$, and $[0{\cdot}4\ \Delta(V_r - V_y)]^2$, necessary for use in the Adams chromatic value formula, are obtained from the new tables, which eliminates the need for a computing machine. The result is given in Adams chromatic value units where $f = 1$. A. H.

Colour Standards for Sugar Cane Syrup and Edible Sugar Cane Molasses. C. B. Broeg and C. F. Walton. *Anal. Chem.*, **24**, 832–834 (May 1952).

A comparison of various methods of colour measurement has led to the recommendation that liquid colour standards be adopted until more permanent and generally accepted standards and methods are evolved. Two stock soln. each containing cobaltous chloride, cupric chloride, and ferric chloride in 10% HCl are made, the concn. of one being three times that of the other, and colour standards are prepared from these by dilution. J. W. D.

Application of Chlorine in the Control of Microbiological Organisms in Process Waters (II p. 357).

Absorption Spectra and Dissociation Constants of Diphenylthiocarbazone (Dithizone) and its Internal Complex Salts—I and II (IV p. 361).

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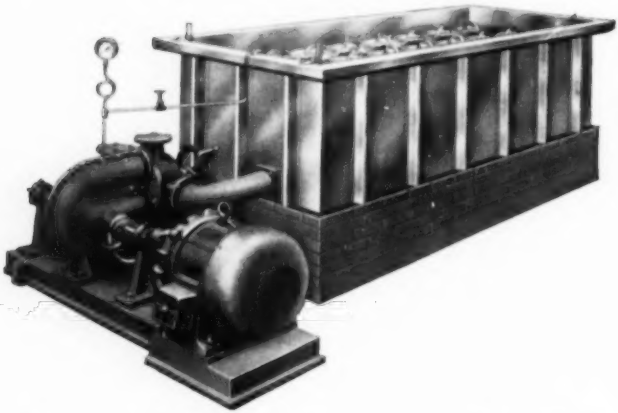

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FORTHCOMING MEETINGS OF THE SOCIETY — *continued*

HUDDERSFIELD SECTION

All meetings held at Field's Cafe, Huddersfield, at 7.30 p.m., unless otherwise stated

1952	
Tuesday 21st Oct.	G. G. Taylor, Esq. and J. C. Brown, Esq. (Clayton Aniline Co.). *Textile Microscopy*
Friday 31st Oct.	ANNUAL DINNER
Monday 3rd Nov.	Dr. C. S. Whewell. *The Future of Textile Finishing* (Joint with Huddersfield Textile Society)
Tuesday 18th Nov.	R. J. Hannay, Esq. (Brotherton & Co.). *The Use of Hydrolysable Esters in the Control of Dyebaths*
Tuesday 2nd Dec.	Sir Wallace Akers, F.R.S. *Chemistry and the Textile Industry* (Joint with R.I.C. Huddersfield Section)
Tuesday 16th Dec.	Professor J. B. Speakman. Title later
1953	
Tuesday 20th Jan.	G. H. Rostron, Esq., A.R.I.C. (J. Crosfield). *Textile Process Soaps*
Tuesday 17th Feb.	INTER-SECTIONAL DISCUSSION
Tuesday 17th March	Dr. H. A. Thomas (Courtaulds). *The Rôle of Dyeing, Printing, and Finishing in Fabric Development*
Tuesday 7th April	ANNUAL GENERAL MEETING followed by a lecture—Dr. J. F. Gaunt. *A Study of the Afterchrome Process of Wool Dyeing*

SCOTTISH SECTION

All meetings at St. Enoch Hotel, Glasgow, 7 p.m., unless otherwise stated

1952	
Tuesday 23rd Sept.	Dr. W. Meitner and H. Hampson, Esq. *Radio-active Isotopes—Continuous Use of Tracers in Printing*
Tuesday 21st Oct.	S. Burgess, Esq. *Economic, Technical and other Difficulties encountered in meeting Fastness Requirements*
Thursday 30th Oct.	Professor J. B. Speakman. Details later
Tuesday 11th Nov.	H. R. Hadfield, Esq. *A Review of the Dyeing of Terylene Polyester Fibre*
Thursday 13th Nov.	J. V. Summersgill, Esq. *Sequestering Agents and their Applications in Textile Processing* W. Beal, Esq. *An Investigation into the Dyeing Properties of Acid and Chrome Dyestuffs and its Practical Significance* This meeting will be held in the Scottish Woollen Technical College, Galashiels, at 7.30 p.m.
Tuesday 9th Dec.	Ministry of Fuel and Power. *Steam Utilisation and Boiler Efficiency*
1953	
Tuesday 13th Jan.	J. C. Brown, Esq. *Microscopy for the Dyer and Finisher*

WEST RIDING SECTION

All meetings held at the Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

1952	
Friday 17th Oct.	SUPPER and SMOKER
Thursday 30th Oct.	A. Gaunt, Esq. and P. Robinson (T. P. Firth & Sons Ltd.). *Dyeing in the Carpet Trade*

WEST RIDING SECTION — *continued*

Thursday 13th Nov.	H. C. Olpin, Esq. and A. J. Wesson, Esq. (British Celanese Ltd.). *Fugitive Tinting with particular reference to Celafibre*
Tuesday 18th Nov.	C. R. M. Ochicke, Esq. (Messrs Newcol). *The Use of Organic Colours in Modern Plastics.* The University, Leeds
Tuesday Nov. 25th	Dr. R. L. Wormell (Courtaulds Ltd.). *Casein and Peanut Protein Fibres* (Joint lecture with the Yorkshire Section of the Textile Institute). Midland Hotel, Bradford
Thursday 27th Nov.	H. R. Hadfield, Esq. (Imperial Chemical Industries Ltd.). *A Review of the Dyeing of Terylene Polyester Fibre*
Thursday 11th Dec.	FILM EVENING. Ladies to be invited. (Joint meeting with Yorkshire Section Textile Institute)
1953	
Friday 16th Jan.	LADIES' EVENING
Thursday 29th Jan.	Lecture. Details later
Thursday 12th Feb.	D. P. Raper Esq. (Imperial Chemical Industries Ltd). *Wool Dyeing Faults and their Correction*
Monday 23rd Feb.	N. R. Hjort, Esq. *Water Treatment.* (Joint lecture with the Halifax Textile Society). Alexandra Cafe, Halifax
Thursday 26th Feb.	J. V. Summersgill Esq. (Geigy Co. Ltd.). Title later
Tuesday 3rd March	Dr. H. Baines (Kodak Ltd.). *Colour Photography.* The University, Leeds
Thursday 12th March	Dr. J. F. Gaunt (Patons & Baldwins Ltd.) *A Study of the Afterchrome Process of Dyeing Wool*
Thursday 26th March	ANNUAL GENERAL MEETING

BRADFORD JUNIOR BRANCH

All meetings held in the Bradford Technical College at 7.15 p.m.

1952	
Tuesday 14th Oct.	J. Brown, Esq. supported by G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Aniline Co. Ltd.). *Uses of Microscopy in Textile Dyeing and Finishing*
Wednesday 29th Oct	Visit to Messrs. Joseph Watson & Sons Ltd., Whitehall Soap Works, Leeds
Friday 14th Nov.	Dr. R. H. Peters (Imperial Chemical Industries Ltd.). *Recent Investigations into the Properties of Vat Dyes*
Thursday 27th Nov.	Visit to Salts (Saltaire) Ltd.
Tuesday 9th Dec.	FILM EVENING
1953	
Monday 19th Jan.	J. W. Fisher, Esq., B.Sc., F.R.I.C. (British Celanese Ltd.). *Synthetic Fibres*
Wednesday 4th Feb.	Visit to Messrs. Montague Burton Ltd., Hudson Road Mills, Leeds
Wednesday 18th Feb.	T. Green, Esq. (Clayton Aniline Co. Ltd.). *The Measurement of pH and its Importance in Relation to Textile Processing*
Thursday 26 Feb.	JUNIOR BRANCH DANCE at the Queens Hall
Friday 13th March	STUDENTS' ANNUAL COMPETITION EVENING
Thursday March 26th	SOCIAL EVENING

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AN appointment will shortly be made to the staff of the British Rayon Research Association of a Textile Technologist who will be required to take charge of a small laboratory to be located at the new headquarters of the British Rayon Federation in London.

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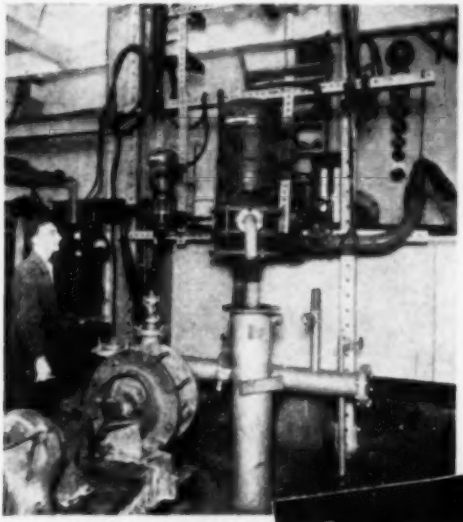

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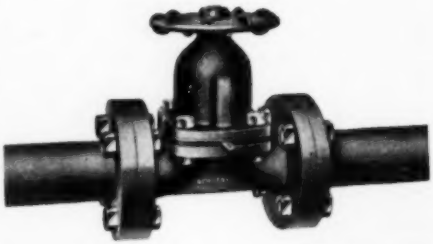

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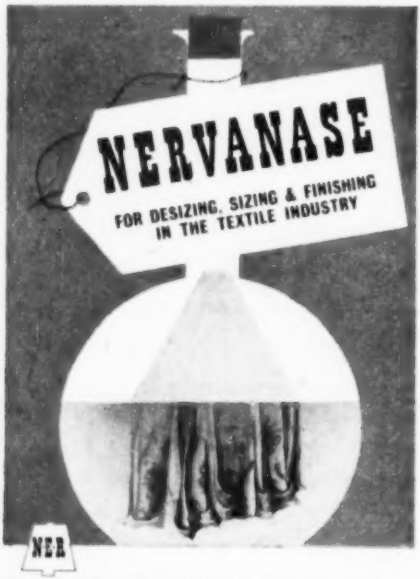

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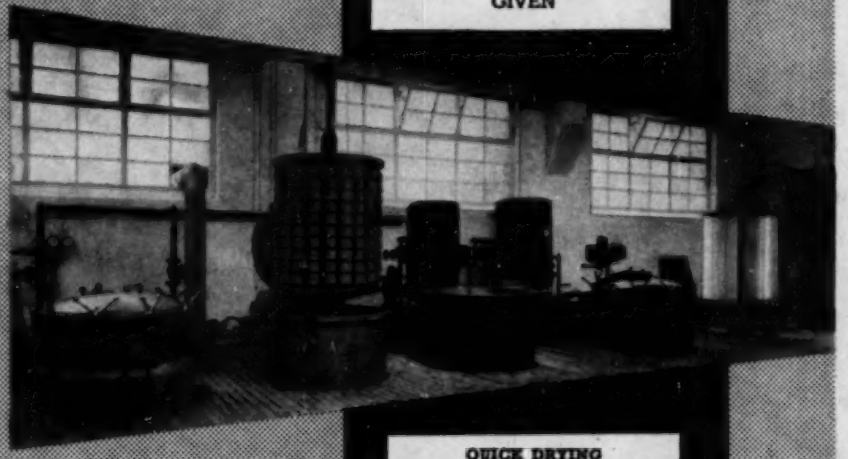

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